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STRENGTH OF HEAT-RESISTANT LAMINATES UP TO 375° C

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SUMMARY

The flexural properties of samples of glass-fabric laminates were determined for several conditions of heating and testing. The laminates tested were bonded with various resins, including unsaturated-polyester, acrylic, silicone, phenolic, and melamine types. Flexural tests were made under the following conditions: (1) At 25° C and 50-percent relative humidity after 200 hours' heating at a temperature T , (2) at a temperature T after heating 0.5 hour at the temperature T , (3) at a temperature T after heating 200 hours at the temperature T , and (4) at 25° C and 50-percent relative humidity without heating prior to testing. The temperature T was 150°, 200°, and 250° C for condition (1); 150°, 200°, 250°, 300°, and 375° C for condition (2); and 150°, 200°, 250°, and 325° C for condition (3).

The one sample of silicone-resin laminate tested was superior to the other laminates in retention of flexural properties at temperatures of 250° C or higher. For conditions (2) and (3) at a temperature of 300° to 325° C this sample retained at least 30 percent of its initial flexural strength and over 50 percent of its initial modulus of elasticity. The single phenolic laminate tested showed good retention of flexural properties when tested at elevated temperatures after 0.5 hour's heating; at 375° C for this condition the flexural strength was 15,000 psi and the modulus of elasticity was about 1,000,000 psi; these values are roughly a third of the initial values (condition (4)). After prolonged heating the behavior of the phenolic sample was much less satisfactory, the strength being almost negligible at 250° C. The sample of melamine laminate was superior to the phenolic in retention of flexural strength after prolonged heating at 250° C; for condition (2) at temperatures above 300° C the melamine was inferior to the phenolic sample. The polyester laminates lost at least 80 percent of their flexural strength when tested at 250° C. For each laminate and each test condition loss in modulus of elasticity correlated with loss in flexural strength, but the former loss was less than the latter.

One test was made in which duplicate sets of specimens of some of the laminates were heated for 200 hours at 200° C; one set was in open bottles in a circulating-air oven and the other set was placed directly on the shelves of the same oven. The phenolic and some of the polyester laminates showed significantly greater losses in flexural strength when

heated on the shelves than when heated in the bottles; no difference was shown by the melamine sample.

INTRODUCTION

The mechanical properties of glass-fabric laminates at elevated temperatures are of interest to aircraft designers as these materials have properties which can be used to advantage in certain applications such as radomes. Considerable work had been done prior to 1947 on the mechanical properties of various laminates at temperatures in the range of 70° to 100° C (references 1 to 7), and a few tests were reported at temperatures of 200° C (reference 8). With the development of higher-speed aircraft and guided missiles there was a demand for information on the mechanical properties of laminated plastics at temperatures much higher than 100° C.

The present investigation was undertaken to determine the flexural properties of promising laminates at temperatures up to 375° C. Commercial resin manufacturers and laminators were informed of the project and invited to submit samples of laminates which could be expected to be heat-resistant. Glass-fabric laminates bonded with various resins including silicone, phenolic, melamine, acrylic, and unsaturated-polyester types were received and tested. This report summarizes the results of the flexural tests made at room temperature after prolonged heating and also at elevated temperatures after both brief and prolonged heating.

Since testing at temperatures of about 300° C presents problems not encountered in testing at room temperatures or at 70° to 100° C, a considerable part of the project was devoted to the development of suitable equipment and procedures. These are described in detail in this report.

This investigation was conducted at the National Bureau of Standards under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics and the Air Materiel Command.

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DEFINITIONS

Following are the flexural properties for a beam of rectangular cross section subjected to a concentrated load P at midspan.

Extreme fiber stress at midspan:

$$\sigma = \frac{3}{2} \frac{PL}{bh^2}$$

where :

P load

L span

b breadth of beam

h depth of beam

Flexural strength:

$$\sigma_{\max} = \frac{3}{2} \frac{P_{\max}L}{bh^2}$$

where $P_{\max}$ is the maximum load and other quantities are as defined previously.

Flexural secant modulus of elasticity for the stress range 0 to σ_1 :

$$E = \frac{L^3}{4bh^3} \frac{P_1}{y_1}$$

where P_1 is the load corresponding to a stress σ_1 , y_1 is the deflection at midspan corresponding to P_1 , and the other quantities are as defined previously.

In statistical terms, the coefficient of variation in percent is:

$$C_v = \frac{\sqrt{\frac{\sum_i (x_i - \bar{x})^2}{N - 1}}}{\bar{x}} 100$$

where

N number of measurements
 x_i i th measurement
 $\bar{x}$ arithmetic mean of x_i 's

MATERIALS AND TEST PROCEDURES

Materials

The materials, submitted in sheets approximately 1/8 inch in thickness, are described in detail in table 1.

Flexural Specimens and Sampling

It is known that the sheet-to-sheet variability, especially in experimental laminates, may be large. A proper sampling plan for the tests at various conditions could give information both as to the average behavior of the sheets of a given sample and as to the variations that might be expected both at standard test conditions and elevated temperatures. Since both the number of sheets and the sizes differed from sample to sample, it was not practical to study sheet-to-sheet variability.

The following sampling plan was adopted: Approximately 80 percent of each sheet of each sample was cut into specimens which were numbered. The numbered specimens were arranged in a random order and separated into groups of seven each. The plan was to test five specimens from each group with two specimens for use in case of experimental difficulties. The statistical analysis of the results can be greatly facilitated by having the same number of test specimens for each condition.

The flexural specimens were 4 inches long and were machined to a width of 1.00 inch either by dry-grinding with a surface grinder or milling with a carbide-tipped cutter.

Flexural Test Equipment¹

The flexural jig and the heated test enclosure are shown in place in the testing machine in figures 1 to 3. The flexural jig and test

¹Drawings of the flexural test equipment are available from the Organic Plastics Section, National Bureau of Standards, Washington 25, D. C.

enclosure are both suspended from the upper cross head of the testing machine. The flexural jig partly disassembled is shown in figure 4. The base of the flexural jig has slots located so that the support blocks may be bolted in place to give a span of 2, 3, 4, or 5 inches. The loading stirrup, attached to the lower cross head of the testing machine (fig. 1), is centered by having the ends of the drill rod used for the loading edge extend into slots in the flexural-jig suspension (see figs. 3 and 4). The flexural jig is made of steel, nickel-plated to avoid corrosion.

The deflection of the specimen is measured by a Southwark-Peters Model PD-1 deflectometer coupled to a recorder on the testing machine to produce load-deflection graphs. The deflectometer, located below the test enclosure (fig. 1), is supported by a platform attached to the flexural-jig base. A glass push rod touching the lower side of the specimen operates the deflectometer; this rod is centered by a guide in the loading stirrup (fig. 4).

The test enclosure has an asbestos-board exterior, a sheet-aluminum interior, and Fiberglas insulation about 1.75 inches thick. The interior is divided into two compartments by an aluminum partition; the front compartment is the test chamber and the rear compartment contains a bank of five 250-watt strip heaters. A fan operating continuously draws air past the heaters and blows it into the test chamber. After a number of tests had been made, it was found desirable to place two layers of 1/4-inch-thick asbestos board in front of the partition (fig. 3) to improve the temperature distribution within the test chamber.

The specimens are inserted with a spatulate holder through a loading port and then placed side by side on an aluminum specimen tray, visible at the right of figure 3. This tray holds five 1-inch-wide specimens. The specimens are manipulated into place in the flexural jig by push rods inserted into holes in the front and right side of the enclosure. In figure 3, one specimen is shown in place in the flexural jig and one in the specimen tray. After a test the specimen is removed from the flexural jig with a pair of tongs inserted through a port in the left side of the enclosure. The operation of the equipment is aided by a double-paned pyrex-glass window and a projection bulb located inside the enclosure. To avoid failure of the phenolic-plastic socket or the bulb, the latter is operated at lower than rated voltage by means of a variable auto transformer (fig. 2) and the socket is attached to the outer wall of the enclosure.

Preliminary temperature measurements were made with thermocouples located inside and on the surface of specimens in both the specimen tray and the flexural jig. It was found that a specimen came to temperature equilibrium within 10 to 15 minutes after insertion in the enclosure. Before the asbestos board was added to the partition, the temperature

of a specimen in the tray was appreciably less than that of a specimen in the flexural jig, the differences amounting to 3° and 7° C at enclosure temperatures of 150° and 250° C, respectively. The difference was reduced to 3° C at 250° C by insertion of the asbestos board.

A fine-mesh screen (fig. 3) was wrapped around the upper part of the flexural jig to keep particles of Fiberglas or resin out of the guides for the loading stirrup.

In testing at elevated temperatures the gaseous products given off by some of the specimens were a source of trouble as the testing machine was located in a controlled-atmosphere room in which the air was recirculated. The problem became more serious as the test temperature was increased. An exhaust pipe (partly visible on the right side of fig. 2) equipped with a blower was finally attached to the box; it was necessary to control the amount of air drawn from the enclosure by means of a valve in the exhaust pipe as otherwise the temperature of the enclosure could not be maintained constant.

Flexural Test Procedure

Temperature conditions.- Sets of specimens of the samples were tested at 25° C after heating at temperatures of 150° , 200° , and 250° C for 200 hours. Other sets of specimens were tested at an elevated temperature T after heating 0.5 hour or 200 hours at the temperature T. The temperatures ranged up to 375° C in the 0.5-hour tests and 325° C in the 200-hour tests. Each sample was not tested at each set of conditions; in some instances the samples were badly weakened at moderate test conditions and in other instances insufficient material was available.

Two control sets of specimens of each sample were tested at 25° C and 50-percent relative humidity. One set was tested at the beginning of the testing program, the other set about 7 months later, near the end of the tests.

In heating the specimens for 200 hours a circulating-air oven was used with each specimen in an open wide-mouthed glass bottle of 2-inch diameter and 4-inch height. In one experiment specimens were also placed on asbestos board laid on the oven shelf in order to compare the effect of heating in still air with heating in rapidly moving air. For convenience, the 200-hour heating period was interrupted at about 96 hours and the specimens were allowed to cool in the glass containers for about 18 hours; the heating was then continued for about 103 hours. The specimens were transferred to the heated test enclosure 1 hour prior to testing; in making the transfer the specimens were transported to the immediate vicinity of the test enclosure in their containers to minimize cooling; transfer from the container into the test enclosure took a few

seconds. During all the 200-hour heating periods the oven temperature was recorded with a Brown recording potentiometer and a thermocouple.

In the tests at elevated temperatures after the specimens were heated 0.5 hour, it was considered that the thermal shock when a cold specimen was placed on the hot metal specimen tray might weaken the specimens. Accordingly, in some tests the specimen tray was covered with a sheet of 1/16-inch-thick asbestos paper.

In tests at 25° C and 50-percent relative humidity, the specimens were normally conditioned for 7 days prior to testing in a room whose atmosphere was controlled at the above values.

Flexural test conditions.- The flexural-jig span was set for 2 inches. The actual span was determined at 25° C with screw micrometer calipers. The contact edges of the supporting and loading pieces were all of 1/4-inch-diameter drill rod. The rate of loading was maintained at 0.04 inch per minute in all tests.

Calculation of flexural data.- In calculations of tests at elevated temperatures the specimen dimensions obtained at 25° C and 50-percent relative humidity were used; however, the span was corrected for change with temperature, assuming a linear thermal expansion coefficient of 12×10^{-6} per degree centigrade for the metal in the jig. The stress range chosen for calculating the values of the flexural secant modulus of elasticity was about one-half to two-thirds of the flexural strength at the test condition concerned.

Resin Content and Loss of Weight on Heating

The resin content was determined for three sets of flexural specimens, except for the laminate made with the silicone resin which could not be completely burned off. The initial weights were determined after the specimens were conditioned for at least 7 days at 25° C and 50-percent relative humidity. The resins were removed from the specimens by heating in a muffle furnace.

The weight loss after 200 hours of heating was determined by weighing some of the sets of specimens prior to heating and reweighing after the specimens were reconditioned 7 days at 25° C and 50-percent relative humidity.

Tolerances on Temperatures and Times of Heating

The temperature in the circulating-air oven was controlled at the nominal value to within $\pm 5^\circ$ C during the 200-hour heating period. In the

heating at 325°C with a muffle furnace, the temperature fluctuations were $\pm 10^{\circ}\text{C}$ relative to 325°C .

In the test chamber the average temperature for each set of five specimens noted at the specimen at the start of the flexural test was within $\pm 3^{\circ}\text{C}$ of the test temperature reported in tables 2 and 3 with only one exception. This exception occurred in testing sample B at approximately 375°C after heating for 0.5 hour, the average test temperature being 368°C . In more than 90 percent of the tests the average test temperature was within 2°C of the desired value. The individual test temperatures were within -5° and 3°C of the nominal values except for the tests at 375°C for which the range was 367° to 381°C .

In the tests at a temperature T after 0.5 hour at the temperature T , the actual time of heating at the start of the flexural test was 30 to 35 minutes. In the 200-hour tests, the total time of heating was 200 ± 1 hours.

RESULTS AND DISCUSSION

The flexural-strength data for the various samples tested under different conditions of temperature and heating are shown in table 2. Some of the data in table 2 are shown graphically in figures 5 to 15. The corresponding data for flexural secant modulus of elasticity are shown in table 3 and figures 16 to 26; the secant moduli selected for graphing are for a stress range from zero to one-half to two-thirds of the flexural strength. The loss-of-weight data for the samples after heating for 200 hours at various temperatures are given in table 4. The precision of the data and other statistical points are discussed in the next section of this report.

In the detailed discussion of the results which follows, it must be noted that, except for the polyester laminates, only one sample of a given type of laminate was tested. Hence only tentative inferences may be drawn regarding the behavior of the various types of materials.

Flexural Strength

The four polyester samples (figs. 5 to 8 and 13) show less than 20-percent loss in strength when tested at 25°C after 200 hours' heating at 150°C . When tested at 25°C after 200 hours' heating at temperatures above 150°C , the loss in strength increases rapidly, reaching about 90 percent at 250°C . These four polyester samples (figs. 14 and 15) show losses in strength ranging from 50 to 90 percent when tested at 150°C after either 0.5 hour's or 200 hours' heating at 150°C . For

similar treatment at 250° C the loss in strength is at least 80 percent, even for samples tested after only 0.5 hour's heating.

The acrylic laminate C, when tested at 150° C after either 0.5 hour's or 200 hours' heating, retained only about 15 percent of its initial strength.

The flexural strength at 25° C of the silicone laminate B was reduced only 10 percent after heating 200 hours at 250° C; this was the least change observed for any sample for this test condition. In tests at elevated temperatures, after heating for either 0.5 hour or 200 hours, the flexural strength dropped considerably between 25° and 150° C. The flexural strength at 150° C was about 13,000 psi compared with a value of 29,000 psi for the controls. Above 150° C the strength diminished very slowly with increase in temperature up to 250° C for the 200-hour and up to 375° C for the 0.5-hour heating period, respectively (fig. 10). It is of interest to compare the results obtained in this laboratory with data obtained by the New York Naval Shipyard (reference 9) on two samples of 0.5-inch-thick glass-fabric silicone laminate from two sources. Some of these results from reference 9 (samples I and II) as well as data for sample B are as follows:

Temperature of conditioning and test (°C)	Flexural strength at T after heating at T (psi)					
	Sample I		Sample II		Sample B	
	Heated 1 hr	Heated 192 hr	Heated 1 hr	Heated 192 hr	Heated 0.5 hr	Heated 200 hr
150	7.2×10^3	17.8×10^3	9.8×10^3	12.0×10^3	12.6×10^3	14.3×10^3
250	3.6	12.8	5.0	4.2	10.9	13.4
Control values	29.9		20.0		29.2	

Sample I was reported to be softened at 250° C when heated only a short time at that temperature; in view of the improvement on further heating, this behavior was believed to have been caused by further cure (reference 9). To test this idea it is reported in reference 9 that specimens of sample I were heated for 72 hours at 250° C, reconditioned for 72 hours at 25° C and 50-percent relative humidity, and then tested at 250° C after 1 to 4 hours at 250° C; flexural strengths of 11,000 psi were obtained.

The sample of phenolic laminate H was outstanding in retention of flexural strength at elevated temperatures when heated only 0.5 hour at the test temperature. For example, the flexural strength at 375° C for this test condition was 15,000 psi compared with 46,000 psi for the control (fig. 11). The same sample, however, was seriously degraded by prolonged heating at 250° C; after such treatment it exhibited a flexural strength much less than 1000 psi when tested either at 250° C or at 25° C.

The melamine laminate J shows a nearly steady decline in strength from 25° to 250° C or more for the three heating and testing conditions. The flexural strength is reduced to about two-thirds, one-half, and one-third for heating at 150°, 200°, and 250° C, respectively. At a given temperature T, the strength at T after 0.5 hour's heating is slightly greater than that for tests at 25° C after 200 hours' heating, and the latter strength is slightly greater than that at a temperature T after 200 hours' heating. These flexural-strength values were compared with data obtained by the New York Naval Shipyard (reference 9), which made tests similar to those described in this report on a 0.5-inch-thick melamine laminate. The general trend of the strength-temperature data given in reference 9 is similar to that shown in figure 12.

Flexural Modulus of Elasticity

For a given sample the trend shown by the graph of flexural secant modulus of elasticity against temperature is quite similar to the plot of flexural strength against temperature for the same condition of heating and testing. However, for each laminate the percentage loss in flexural modulus of elasticity for a given test condition is always less than the loss in flexural strength for that test condition.²

Correlation between Changes in Flexural Strength and Flexural Modulus of Elasticity

In figure 27 the flexural strength in percent of control is plotted against the flexural secant modulus of elasticity in percent of control for all the laminates except C. Data for all conditions in tables 2 and 3 were used except that the values for special conditions were not plotted unless no other data at a given temperature were available. The modulus-of-elasticity data for the lowest stress range were used in the case of tests at 150° C. It is evident that a correlation exists between these two quantities; furthermore, it appears that the correlation equation is probably different for the polyester laminates as compared with

²There were exceptions to this statement when the loss in the flexural property was 5 percent or less. Such losses are not considered significant.

that of the melamine laminate J. A detailed analysis of the correlation was not made as only in the case of the polyester laminates was there more than one sample of a given type of laminate.

Relative Heat Resistance of the Laminates

To give a concise comparison of the laminates with regard to their retention of flexural properties at elevated temperatures, a table was prepared in the following manner: For each sample the temperatures in figures 5 to 12 were noted for which the flexural strength was 30 percent or more of the flexural strength for the control for all three conditions of heating and testing. The same procedure was followed for the corresponding values of secant modulus of elasticity in figures 16 to 23 except that the percentage was 50 instead of 30. These temperature limits are shown in table 5. Since it was often necessary to interpolate the stress range as well as the modulus of elasticity, the temperatures given in table 5 for the modulus data are less accurate than the temperatures for the flexural strength. It must be emphasized in addition that, for the modulus-of-elasticity data, temperatures different from those given in table 5 would apply for other stress ranges; when the stress-strain diagram is nearly linear for the ranges considered, the effect of differing stress range is slight.

It is of interest to compare the values in table 5 with data obtained by Sieffert and Schoenborn (reference 10) who determined the weight loss as a function of time for a number of laminates at various temperatures; from these data curves were plotted of weight loss against temperature. It was found that for each material the weight loss as a function of temperature began to increase rapidly beyond a certain temperature. By using an arbitrary criterion M, a "critical thermal instability temperature" was determined from the plotted data. The values Sieffert and Schoenborn reported for several laminates are as follows:

Glass-fabric laminate	Resin content (percent)	Critical thermal instability temperature; M = 4, 72-hr data (°C)	Temperature for 5-percent resin loss; 72-hr data (°C)
Polyester	31	140	140
Silicone A	52	350	375
Silicone B	40	355	250
Low-pressure phenolic	28	210	220
Melamine-formaldehyde A	40	235	115
Melamine-formaldehyde B	45	240	100

It is evident that there is close agreement between the critical thermal instability temperature and the temperatures given in table 5 for retention of 30 percent of original flexural strength.

Effect of Speed of Heated Air on Changes in Flexural Properties

The data in tables 2 and 3 for tests at 25° C after heating the specimens at 200° C either on the oven shelf or in open bottles were examined statistically (see Statistical Analysis). It was found that both the flexural strengths and flexural moduli of elasticity for the polyester laminates and the phenolic laminate were significantly lower for the case of heating on the oven shelf, that is, in rapidly moving air, as compared with heating in the open bottles. However, for the melamine laminate J the differences were not significant.

These results indicate that information on the effect of prolonged heating on the glass-fabric laminates under different atmospheric conditions is needed. The degradation of the resin may depend on the rate of removal of degradation products as well as on the rate at which heated air containing oxygen, an important agent for some degradation reactions, is swept over the surface of the material. It follows that data of the type presented in this report on the properties of laminates after prolonged heating should be used for design purposes with caution since in practice the conditions of heating may differ widely from those that apply to the data given here. Likewise, interlaboratory comparisons of a given laminate may give discordant results if the heating conditions are not duplicated.

Loss of Weight on Heating

After 200 hours' heating at only 150° C all the samples except the silicone exhibited losses in resin of 2 to 8 percent of the initial amount, with the melamine laminate showing the greatest loss. As the temperature of heating was raised, the loss in amount of resin increased very rapidly for all the polyester laminates and the one phenolic laminate. For these samples the resin loss is roughly fourfold at 200° C and twelvefold to thirtyfold at 250° C, compared with the losses at 150° C. In contrast with this behavior the melamine laminate showed much less rapid increases in resin loss (see table 4).

STATISTICAL ANALYSIS

Precision of Results

The standard deviation was determined for the data in tables 2 and 3 for tests at various conditions. For a given sample it was observed that the standard deviation tended to be proportional to the mean value. For this reason further analysis was made with the coefficient of variation instead of the standard deviation.

The coefficient of variation was not reported for each value of flexural strength in table 2 or each value of modulus of elasticity in table 3 because such statistics, based on only five observations, are subject to wide variability. It was decided to analyze the individual coefficient of variation with respect to its variation from sample to sample and between the different temperatures. This was done to determine in what manner these data could best be combined for more reliable estimates of precision. The analyses are discussed in the following paragraphs.

Flexural strength.- The coefficient of variation C_v was determined for all the values in table 2. A statistical analysis of some of these data indicates some variation of the value of C_v between samples. No consistent trend for the effect of conditioning or test temperature on the value of C_v for the samples was noted. Therefore, the estimates of C_v within each sample were combined with the following results:

Laminate	C_v (percent)
Unsaturated polyester	
A	6.6
E	5.1
F	7.3
G	8.4
Acrylic addition	
C	8.8
Silicone	
B	5.2
Phenolic	
H	10
Melamine	
J	2.9

In a few instances, usually for the highest testing or conditioning temperature and when the flexural strength was a small fraction of the initial value, a very large value of C_v was obtained; such values were omitted in obtaining the above estimates of C_v .

Flexural modulus of elasticity. - The coefficient of variation was determined for the modulus values in table 3. A statistical analysis was carried out for the tests made either at 25° C or at a temperature T after 200 hours' heating at T. It was found that (1) the effect of conditioning temperature on C_v varies significantly from sample to sample, (2) C_v cannot be assumed to be the same for all samples, and (3) the effect of testing temperature, that is, 25° C compared with T, on C_v varies significantly from sample to sample. The above effects do not seem to be important except the sample-to-sample variability. It was therefore decided to combine the estimates of C_v for the various samples. The estimated values of C_v for the modulus of elasticity are:

Laminate	C_v (percent)
Unsaturated polyester	
A	7.5
E	5.1
F	8.8
G	14
Acrylic addition	
C	7
Silicone	
B	4.5
Phenolic	
H	10
Melamine	
J	3.0

In a few instances, usually for the highest testing or conditioning temperature and when the modulus of elasticity was a small fraction of the initial value, a very large value of C_v was obtained; such values were omitted in obtaining the above estimates of C_v .

Thermal Shock in Tests after 0.5 Hour's Heating

A statistical analysis was made of the flexural data for the tests after 0.5 hour's heating in which duplicate sets of specimens were tested, one set with and one set without asbestos paper on the metal specimen tray. All the samples did not behave alike with respect to differences in flexural strength or modulus of elasticity for the two tray conditions. While in some cases there appears to be a significant effect, the data as a whole do not support the assumption that the thermal shock is serious.

Effect of Heating in Still Air Compared with Heating in Rapidly Moving Air

A statistical analysis was made of the flexural data for the tests at 25° C after 200 hours' heating at 200° C with the specimens exposed (1) in open bottles and (2) on the oven shelf. It was found that flexural strength and flexural modulus of elasticity were similarly affected for specimens contained in open bottles compared with those placed directly on the oven shelf during the heating period. The data for each sample were tested separately. Samples A, G, and H all show significantly greater losses in flexural strength and modulus of elasticity for shelf heating compared with the losses for bottle heating. For sample E the effect was significant for flexural strength but not for modulus of elasticity. For sample J no significant effect was noted.

SUMMARY OF RESULTS

For several conditions of heating and testing, the results of tests to determine the flexural properties of samples of glass-fabric laminates bonded with various resins may be summarized as follows. It should be noted that these results, except for those of the polyester laminates, are based on data for only one sample of each type of glass-fabric laminate and hence can be considered only tentatively as being representative of the various types of material.

1. The silicone laminate was superior to the other laminates tested in retention of flexural properties at temperatures of 250° to 300° C. When tested at about 325° C after either 0.5 hour's or 200 hours' heating at that temperature, this laminate retained at least 30 percent of its initial flexural strength and over 50 percent of its initial flexural modulus of elasticity.

2. The phenolic laminate showed good retention of its flexural properties when tested at elevated temperatures after heating for 0.5 hour at the test temperature. When tested in this way at 375° C, the flexural

strength was 15,000 psi, and the modulus of elasticity was 1.0×10^6 psi, values roughly one-third of the corresponding values for the controls. The behavior of the phenolic laminate after prolonged heating was much less satisfactory; at 25° or 250° C after 200 hours' heating at 250° C the flexural strength was much less than 1000 psi.

3. The melamine laminate, although much inferior to the phenolic laminate when tested at a temperature of 375° C after 0.5 hour's heating, was superior to the phenolic in retention of flexural strength after 200 hours' heating at 250° C.

4. The four polyester laminates lost at least 80 percent of their flexural strengths when tested at 250° C after either 0.5 hour's or 200 hours' heating.

5. The acrylic addition laminate lost at least 80 percent of its flexural strength when tested at 150° C after either 0.5 hour's or 200 hours' heating.

6. For each laminate and each test condition, loss in flexural modulus of elasticity correlated with loss in flexural strength; the former loss was less than the latter.

7. The loss in flexural properties during prolonged heating may depend on the method of exposing the specimens in the oven. For specimens of the polyester and phenolic laminates, heating in a circulating-air oven in open bottles caused significantly less loss in flexural properties than did heating on the shelves of the oven.

National Bureau of Standards

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TABLE 1.- DESCRIPTION OF MATERIALS

Sample	A	E	F	G	C	B	H	J
Laminator of sample	American Cyanamid Co.	Plaskon Division, Libby-Owens-Ford Glass Co.	Monsanto Chemical Co.	Rohm & Haas Co., Inc.	E. I. du Pont de Monsieur & Co., Inc.	Dow Corning Corp.	Rohm & Haas Co., Inc.	Formica Insulation Co.
Resin used: Trade name and number	Laminate PDL-7-261, B-11	Plaskon 911-11	Thalid X-588-10A.C.	Paraplex P-43	ECM monomer, 60 per- cent; Polyvinyl methacrylate BM-4, 40 percent	DC 804	Redux	Melmac 405
Chemical type	Unsaturated polyester	Unsaturated polyester	Unsaturated polyester; vinyl-type monomers	Unsaturated polyester-styrene	Acrylic addition type; thermosetting	Silicone	Phenolic	Melamine-formaldehyde
Catalyst used with resin: Name or type	Lupercol ATC ¹	Lupercol ATC ¹	Lupercol ATC ¹	Benzoyl peroxide	Residual benzoyl per- oxide in polyvinyl methacrylate	-----	None	None
Amount, percent	0.5	4	2	1	-----	-----	-----	-----
Liquid added to resin to prepare it for applica- tion to fabric: Name	Styrene	None	None	Styrene	Methyl ethyl ketone	Toluene	None	Water, 95 percent; butyl alcohol, 5 percent
Amount, parts per 100 parts of resin	40	-----	-----	15	135	67	-----	67
Resin content (by weight) of finished panel: Manufacturer's data, percent	33 ± 2	31	45	40-45	56	39	40-45	42-47
National Bureau of Standards data, percent	35	32	# 24	42	62	--	44	46
Fabric used: Identity	Fiberglass 181-A-14	Fiberglass 181-A-14	O.C.C. 181-A-14	Fiberglass 181-38, finish 114	Fiberglass 181-A-13	Fiberglass 181-A-12	Fiberglass 181-38, finish 114	Fiberglass EDC-128, sizing pyrolyzed to light brown
Number of plies	12	15	15	12	6	11	12	13
Play arrangement	Parallel	Parallel	Cross	Cross	Cross	Cross	Cross	Parallel
Treatment prior to application of resin (drying, etc.)	None	In forced-draft oven for 2 hr at 204° C	None	None	None	-----	None	-----
Method of applying resin	Dipping	By hand with broad- blade spatula	Brush	Brush	Continuous dip and squeeze roll	Dipping	Dipping	Dipping
Drying of impregnated fabric	-----	-----	-----	-----	Solvent evaporated in forced-draft oven at 43° C	-----	Air-dried for 30 min at room temperature; 15-18 min at 104° C in forced-draft oven	-----
Molding conditions: Equipment	Steam Hydraulic press	Steam Hydraulic press	Steam Platen press	Steam Hydraulic press	Steam Hydraulic press	Steam Hydraulic press	Steam Hydraulic press	Steam Hydraulic press
Pressure	None	None	None	104	None	110	145	None
Temperature, °C	105, 15 min	100	100-110	145	110	175	145	25-124, 20 min 142-147, 20 min 1100
Pressure, psi	125, 10 min	25	15	50	50	900	50	-----
Time, min	10	30	15	5	15	75	15	About 40
Cure	None	None	5	None	3	90° C, 15 hr ² 130° C, 4 hr 160° C, 4 hr 190° C, 16 hr 250° C, 4 hr	None	15 None
Time of heating under pressure, min	25	None	None	None	None	-----	-----	-----
Time of cooling under pressure, min	None	None	None	None	None	-----	-----	-----
Aftercure	None	None	None	None	None	-----	-----	-----
Description of sample:	12 by 22 and 21 by 22	12 by 12	12 by 12	10 by 10	14 by 19 and 11 by 12	12 by 12	10 by 10	21 by 36
Area, in.	0.11-0.13	0.13-0.14	0.09-0.12	0.13-0.15	0.10-0.13	0.13-0.14	0.13-0.15	0.12-0.13
Thickness, in.	1 of each	10	4	14	3 of first area; 2 of second	3	14	1
Number of sheets	1 of each	10	4	14	3 of first area; 2 of second	3	14	1

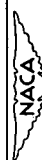
¹Lupercol ATC consists of 50 percent benzoyl peroxide and 50 percent tricresyl phosphate.²Mechanical convection oven.

TABLE 2.- FLEXURAL-STRENGTH DATA FOR SPECIMENS OF CLASS-FABRIC LAMINATES TESTED UNDER VARIOUS CONDITIONS OF TEMPERATURE AND HEATING

[Specimens approx. 1/8 in. thick and 1 in. wide tested at a relative head motion of 0.04 in./min with a span of 2 in. Each value is average for five specimens unless otherwise noted. Specimens taken at random from sheets unless otherwise noted.]

Temperature of heating (°C)	Special conditions	Unsat. polyester-resin laminate A				Unsat. polyester-resin laminate E				Unsat. polyester-resin laminate F				Unsat. polyester-resin laminate G			
		Mean (psi)	Range (psi)	Percent of control mean		Mean (psi)	Range (psi)	Percent of control mean		Mean (psi)	Range (psi)	Percent of control mean		Mean (psi)	Range (psi)	Percent of control mean	
Flexural strength of control specimens at 25° C after at least 7 days at 25° C and 50-percent relative humidity																	
None	A ^a	27.1 × 10 ³	24.6-29.9 × 10 ³	---		58.8 × 10 ³	56.6-61.8 × 10 ³	---		49.8 × 10 ³	47.1-52.4 × 10 ³	---		53.7 × 10 ³	47.1-64.4 × 10 ³	---	
Do----	B ^a	27.9	25.0-31.2	---		59.2	56.2-60.6	---		50.8	48.4-52.9	---		51.3	49.5-53.4	---	
Do----	Control mean	27.5				59.0				50.3				52.5			
Flexural strength of specimens at 25° C after 200-hr heating ^b (reconditioned 7 days at 25° C and 50-percent relative humidity, unless otherwise noted)																	
150		25.8 × 10 ³	23.4-28.1 × 10 ³	94		57.7 × 10 ³	51.0-63.1 × 10 ³	98		49.6 × 10 ³	48.1-51.7 × 10 ³	99		43.6 × 10 ³	40.1-49.3 × 10 ³	83	
150	Not reconditioned ^c	28.4	26.0-30.9	103		60.2	56.5-64.4	102		52.1	49.7-54.7	104		41.1	37.6-46.9	78	
200		20.4	17.9-22.3	74		40.8	35.7-44.9	69		---	---	---		26.6	24.4-30.3	31	
200	Not heated in bottles ^d	17.8	16.7-19.4	65		37.0	34.4-40.5	63		---	---	---		21.4	18.3-24.3	41	
250		3.6	3.3-4.1	13		9.6	7.3-10.6	16		3.5	2.9-4.2	7		3.0	2.8-3.4	6	
Flexural strength of specimens at a temperature T after 0.5-hr heating at temperature T																	
150		9.2 × 10 ³	8.4-9.6 × 10 ³	33		20.1 × 10 ³	18.0-21.2 × 10 ³	34		12.0 × 10 ³	11.0-12.8 × 10 ³	24		6.2 × 10 ³	4.9-7.4 × 10 ³	12	
200		6.1	5.8-6.3	22		10.7	9.9-11.4	18		7.4	6.4-8.9	15		4.9	4.3-6.3	9	
250		2.4	4.7-6.2	20		7.7	7.1-8.0	13		5.8	5.3-6.6	12		3.6	3.3-4.1	7	
250	Asbestos-covered tray ^e	5.0	4.7-5.4	18		7.8	7.4-8.4	13		---	---	---		4.0	3.6-4.6	8	
300		3.5	3.2-3.9	13		5.4	5.2-5.6	9		4.4	3.1-5.1	7		---	---	---	
300	Asbestos-covered tray ^e	3.2	3.1-3.4	12		5.5	5.2-5.9	9		5.3	4.7-5.7	11		---	---	---	
375	Asbestos-covered tray ^e	---	---	---		---	---	---		---	---	---		---	---	---	
Flexural strength of specimens at a temperature T after 200-hr heating at temperature T ^b																	
150		14.7 × 10 ³	14.5-14.8 × 10 ³	53		28.9 × 10 ³	27.2-30.6 × 10 ³	49		24.1 × 10 ³	23.6-25.0 × 10 ³	48		7.1 × 10 ³	6.2-7.9 × 10 ³	14	
200		6.4	6.2-6.7	23		18.2	17.1-19.4	31		17.4	16.6-18.3	35		3.4	3.3-3.5	6	
250		2.1	1.7-2.3	8		9.4	8.6-10.1	16		2.2	2.1-2.4	4		---	---	---	
325	(g)	---	---	---		---	---	---		---	---	---		---	---	---	

^aA, first set of controls (Oct. 1943); B, second set of controls (April 1949).

Unless otherwise noted, all specimens which were heated for 200 hr were heated in open wide-mouthed bottles placed in a circulating-air oven. After removal from oven, specimens kept over calcium chloride for 24 hr prior to testing.

^bSpecimens laid on asbestos board on oven shelf.

^cNormal tray is an aluminum tray. Asbestos-covered tray is same aluminum tray covered with asbestos paper about 1/16 in. thick. Unless otherwise noted, all tests at elevated temperature were made with normal tray.

^dSpecimens not randomly selected but taken from at least three sheets.

^eSpecimens not randomly selected but taken from at least three sheets.

^fSpecimens not randomly selected but taken from at least three sheets.

^gA muffle furnace controlled at 325° ± 10° C was used for conditioning these specimens.



TABLE 2.- FLEXURAL-STRENGTH DATA FOR SPECIMENS OF GLASS-FABRIC LAMINATES TESTED UNDER VARIOUS CONDITIONS OF TEMPERATURE AND HEATING - Concluded

Temperature of heating (°C)	Special conditions	Acrylic-resin laminate C			Silicone-resin laminate B			Phenolic-resin laminate H			Melamine-resin laminate J		
		Mean (psi)	Range (psi)	Percent of control mean (h)	Mean (psi)	Range (psi)	Percent of control mean	Mean (psi)	Range (psi)	Percent of control mean	Mean (psi)	Range (psi)	Percent of control mean
Flexural strength of control specimens at 25° C after at least 7 days at 25° C and 50-percent relative humidity													
None	A ^a	24.3 × 10 ³	23.7-25.1 × 10 ³	-----	28.9 × 10 ³	28.2-29.7 × 10 ³	---	44.9 × 10 ³	35.0-50.6 × 10 ³	---	51.6 × 10 ³	50.2-53.4 × 10 ³	---
Do-----	B ^a	26.8	25.1-28.4	-----	29.6	27.7-31.3	---	48.2	42.0-53.6	---	51.5	50.0-52.8	---
Do-----	Control mean	-----	-----	-----	29.2	-----	---	46.5	-----	---	51.6	-----	---
Flexural strength of specimens at 25° C after 200-hr heating ^b (reconditioned 7 days at 25° C and 50-percent relative humidity, unless otherwise noted)													
150	-----	28.2 × 10 ³	26.8-29.4 × 10 ³	115(A) ^a	27.7 × 10 ³	26.2-30.3 × 10 ³	95	40.1 × 10 ³	36.4-42.6 × 10 ³	86	33.8 × 10 ³	31.8-35.0 × 10 ³	66
150	Not reconditioned ^c	31.7	27.6-34.9	130(A) ^a	28.0	26.6-29.1	96	40.3	36.3-43.3	87	31.6	31.3-32.1	61
200	-----	-----	-----	-----	-----	-----	---	15.0	13.2-16.8	32	26.4	25.7-28.1	91
200	Not heated in bottles ^d	-----	-----	-----	-----	-----	---	9.5	8.6-10.4	20	26.0	25.1-26.8	90
250	-----	-----	-----	-----	26.4	25.2-27.7	90	<2	-----	---	17.6	16.8-18.4	34
Flexural strength of specimens at a temperature T after 0.5-hr heating at temperature T													
150	-----	3.4 × 10 ³	2.9-4.2 × 10 ³	14(A) ^a	12.6 × 10 ³	11.3-13.7 × 10 ³	43	41.9 × 10 ³	39.7-44.4 × 10 ³	90	37.2 × 10 ³	36.4-38.1 × 10 ³	72
200	-----	-----	-----	-----	10.9	10.1-11.9	37	35.3	28.2-40.6	76	28.5	24.8-27.4	51
250	Asbestos-covered tray ^e	4.7	4.2-5.1	1.8(B) ^a	10.1	9.6-10.9	135	24.8	21.5-28.7	51	22.1	21.4-22.7	43
300	-----	-----	-----	-----	10.5	10.3-10.8	32	19.8	17.4-21.7	23	20.5	21.8-23.2	40
300	Asbestos-covered tray ^f	-----	-----	-----	10.5	10.3-10.8	36	14.7	12.0-22.3	44	19.0	19.2-22.6	37
375	Asbestos-covered tray ^g	-----	-----	-----	19.8	18.8-20.6	34	14.7	12.3-16.4	32	2.0	1.4-2.5	14
Flexural strength of specimens at a temperature T after 200-hr heating at temperature T ^h													
150	-----	4.2 × 10 ³	2.9-5.4 × 10 ³	17(A) ^a	14.3 × 10 ³	13.4-15.7 × 10 ³	49	39.1 × 10 ³	36.2-42.6 × 10 ³	84	30.3 × 10 ³	29.4-32.3 × 10 ³	59
200	-----	-----	-----	-----	13.4	12.7-14.0	46	10.9	10.4-11.9	23	23.3	23.0-24.2	45
250	-----	-----	-----	-----	9.8	9.2-10.4	34	5.55	5.1-5.9	1.2	15.6	14.7-17.4	30
325	(g)	-----	-----	-----	-----	-----	---	-----	-----	---	51.5	51.5-1.6	33

^aA, first set of controls (Oct. 1948); B, second set of controls (April 1949).

^bUnless otherwise noted, all specimens which were heated for 200 hr were heated in open wide-mouthed bottles placed in a circulating-air oven.

^cAfter removal from oven, specimens kept over calcium chloride for 24 hr prior to testing.

^dSpecimens laid on asbestos board on oven shelf.

^eNormal tray is an aluminum tray. Asbestos-covered tray is same aluminum tray covered with asbestos paper about 1/16 in. thick. Unless otherwise noted, all tests at elevated temperature were made with normal tray.

^fSpecimens not randomly selected but taken from at least three sheets.

^gA muffle furnace controlled at 325° ± 10° C was used for conditioning these specimens.

^hFlexural strengths for first and second controls differ significantly for this laminate. Test results for various conditions were referred to data for control tested at nearly the same time; this is indicated by (A) or (B) following the percentage.

ⁱFour specimens.

^jTwo specimens.

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TABLE 3.- SECANT MODULUS OF ELASTICITY FOR SPECIMENS OF GLASS-FABRIC LAMINATES TESTED UNDER VARIOUS CONDITIONS OF TEMPERATURE AND HEATING

[Specimens approx. 1/8 in. thick and 1 in. wide tested at a relative head motion of 0.04 in./min with a span of 2.0 in. Each value is average for five specimens unless otherwise noted. Specimens taken at random from sheets unless otherwise noted.]

Temperature of heating (°C)	Special conditions	Unsat. polyester-resin laminate A				Unsat. polyester-resin laminate E				Unsat. polyester-resin laminate F				Unsat. polyester-resin laminate G			
		Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	
Modulus of elasticity of control specimens at 25° C after at least 7 days at 25° C and 50-percent relative humidity																	
σ = 0 to 2500 psi																	
None	A ^b	2.72 × 10 ⁶	2.52-2.85 × 10 ⁶	---	3.43 × 10 ⁶	3.34-3.48 × 10 ⁶	---	3.87 × 10 ⁶	3.77-3.97 × 10 ⁶	---	2.68 × 10 ⁶	2.34-3.24 × 10 ⁶	---	2.68 × 10 ⁶	2.34-3.24 × 10 ⁶	---	---
Do----	B ^b	2.80	2.61-3.19	---	3.38	3.29-3.56	---	3.86	3.76-3.96	---	2.54	2.41-2.60	---	2.54	2.41-2.60	---	---
Do----	Control mean	2.76	-----	---	3.41	-----	---	3.87	-----	---	2.61	-----	---	2.61	-----	---	---
σ = 0 to 4000 psi																	
Do----	A ^b	2.71 × 10 ⁶	2.52-2.85 × 10 ⁶	---	3.43 × 10 ⁶	3.34-3.48 × 10 ⁶	---	3.85 × 10 ⁶	3.71-3.91 × 10 ⁶	---	2.68 × 10 ⁶	2.34-3.24 × 10 ⁶	---	2.68 × 10 ⁶	2.34-3.24 × 10 ⁶	---	---
Do----	B ^b	2.80	2.61-3.19	---	3.34	3.25-3.50	---	3.83	3.72-3.96	---	2.51	2.41-2.60	---	2.51	2.41-2.60	---	---
Do----	Control mean	2.75	-----	---	3.39	-----	---	3.84	-----	---	2.60	-----	---	2.60	-----	---	---
σ = 0 to 5000 psi																	
Do----	A ^b	2.70 × 10 ⁶	2.52-2.85 × 10 ⁶	---	3.43 × 10 ⁶	3.34-3.48 × 10 ⁶	---	3.69 × 10 ⁶	3.56-3.82 × 10 ⁶	---	2.67 × 10 ⁶	2.34-3.24 × 10 ⁶	---	2.67 × 10 ⁶	2.34-3.24 × 10 ⁶	---	---
Do----	B ^b	2.80	2.61-3.19	---	3.30	3.20-3.49	---	3.70	3.64-3.83	---	2.50	2.40-2.60	---	2.50	2.40-2.60	---	---
Do----	Control mean	2.75	-----	---	3.37	-----	---	3.69	-----	---	2.59	-----	---	2.59	-----	---	---
σ = 0 to 8000 psi																	
Do----	A ^b	2.69 × 10 ⁶	2.52-2.85 × 10 ⁶	---	3.36 × 10 ⁶	3.25-3.40 × 10 ⁶	---	---	---	---	2.63 × 10 ⁶	2.32-3.19 × 10 ⁶	---	2.63 × 10 ⁶	2.32-3.19 × 10 ⁶	---	---
Do----	B ^b	2.79	2.54-2.97	---	3.22	3.07-3.44	---	---	---	---	2.46	2.37-2.57	---	2.46	2.37-2.57	---	---
Do----	Control mean	2.74	-----	---	3.29	-----	---	---	---	---	2.55	-----	---	2.55	-----	---	---
σ = 0 to 10,000 psi																	
Do----	A ^b	2.68 × 10 ⁶	2.53-2.85 × 10 ⁶	---	---	---	---	---	---	---	2.59 × 10 ⁶	2.27-3.15 × 10 ⁶	---	2.59 × 10 ⁶	2.27-3.15 × 10 ⁶	---	---
Do----	B ^b	2.78	2.56-3.17	---	---	---	---	---	---	---	2.42	2.26-2.54	---	2.42	2.26-2.54	---	---
Do----	Control mean	2.73	-----	---	---	---	---	---	---	---	2.51	-----	---	2.51	-----	---	---
σ = 0 to 20,000 psi																	
Do----	A ^b	2.58 × 10 ⁶	2.45-2.80 × 10 ⁶	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Do----	B ^b	2.60	2.38-2.84	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Do----	Control mean	2.59	-----	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Modulus of elasticity of specimens at 25° C after 200-hr heating ^d (reconditioned 7 days at 25° C and 50-percent relative humidity, unless otherwise noted)																	
σ = 0 to 10,000 psi																	
150	Not reconditioned ^e	2.60 × 10 ⁶	2.38-2.91 × 10 ⁶	95	3.14 × 10 ⁶	3.11-3.18 × 10 ⁶	93	3.56 × 10 ⁶	3.51-3.67 × 10 ⁶	93	2.40 × 10 ⁶	2.33-2.47 × 10 ⁶	93	2.40 × 10 ⁶	2.33-2.47 × 10 ⁶	93	93
150		Do----	2.70	2.54-2.97	99	3.30	3.13-3.40	98	3.70	3.59-3.86	96	2.36	2.23-2.50	91	2.36	2.23-2.50	91
σ = 0 to 20,000 psi																	
150	Not reconditioned ^e	2.54 × 10 ⁶	2.31-2.84 × 10 ⁶	98	3.08 × 10 ⁶	3.02-3.17 × 10 ⁶	94	3.29 × 10 ⁶	3.23-3.39 × 10 ⁶	89	2.37 × 10 ⁶	2.28-2.46 × 10 ⁶	93	2.37 × 10 ⁶	2.28-2.46 × 10 ⁶	93	93
150		Do----	2.67	2.51-2.87	103	3.23	3.08-3.35	98	3.46	3.38-3.59	94	2.29	2.17-2.45	90	2.29	2.17-2.45	90
σ = 0 to 10,000 psi																	
200	Not heated in bottles ^f	2.43 × 10 ⁶	2.22-2.66 × 10 ⁶	89	2.82 × 10 ⁶	2.75-2.92 × 10 ⁶	84	---	---	---	2.12 × 10 ⁶	1.75-2.65 × 10 ⁶	82	2.12 × 10 ⁶	1.75-2.65 × 10 ⁶	82	82
200		Do----	2.21	2.02-2.38	81	2.73	2.60-2.98	81	---	---	---	1.55	1.34-1.85	60	1.55	1.34-1.85	60
σ = 0 to 2500 psi																	
250	Do----	1.38 × 10 ⁶	1.10-1.55 × 10 ⁶	50	1.62 × 10 ⁶	1.39-1.89 × 10 ⁶	48	2.16 × 10 ⁶	1.72-2.54 × 10 ⁶	56	0.91 × 10 ⁶	0.67-1.25 × 10 ⁶	35	0.91 × 10 ⁶	0.67-1.25 × 10 ⁶	35	35

^aReferred to control mean secant modulus for corresponding stress range; in cases where the least-stress range, 0 to σ₁ psi, for the control specimens is higher than the stress range for the heated specimens, the stress-deflection graph was linear within the experimental accuracy below the stress σ₁.

^bA, first set of controls (Oct. 1948); B, second set of controls (April 1949).

^cInterpolated value.

^dUnless otherwise noted, all specimens which were heated for 200 hr were heated in open wide-mouthed bottles placed in a circulating-air oven.

^eAfter removal from oven, specimens kept over calcium chloride for 24 hr prior to testing.

^fSpecimens laid on asbestos board on oven shelf.



TABLE 3.- SECANT MODULUS OF ELASTICITY FOR SPECIMENS OF GLASS-FABRIC LAMINATES TESTED UNDER VARIOUS CONDITIONS OF TEMPERATURE AND HEATING - Continued

Temperature of heating (°C)	Special conditions	Unsat. polyester-resin laminate A				Unsat. polyester-resin laminate E				Unsat. polyester-resin laminate F				Unsat. polyester-resin laminate G			
		Mean (psi)	Range (psi)	Percent of control mean (a)		Mean (psi)	Range (psi)	Percent of control mean (a)		Mean (psi)	Range (psi)	Percent of control mean (a)		Mean (psi)	Range (psi)	Percent of control mean (a)	
Modulus of elasticity of specimens at a temperature T after 0.5-hr at the temperature T																	
σ = 0 to 5000 psi																	
150	-----	1.48 × 10 ⁶	1.24-1.66 × 10 ⁶	54		2.50 × 10 ⁶	2.34-2.99 × 10 ⁶	73		2.84 × 10 ⁶	2.74-2.99 × 10 ⁶	73		0.86 × 10 ⁶	0.63-1.07 × 10 ⁶	33	
σ = 0 to 8000 psi																	
150	-----	1.27 × 10 ⁶	1.04-1.40 × 10 ⁶	46		2.44 × 10 ⁶	2.32-2.92 × 10 ⁶	72		2.46 × 10 ⁶	2.15-2.68 × 10 ⁶	64		0.80 × 10 ⁶	0.61-0.97 × 10 ⁶	31	
σ = 0 to 4000 psi																	
200	-----	1.23 × 10 ⁶	1.09-1.37 × 10 ⁶	45		1.72 × 10 ⁶	1.62-1.81 × 10 ⁶	50		1.88 × 10 ⁶	1.69-2.35 × 10 ⁶	49		0.84 × 10 ⁶	0.63-1.32 × 10 ⁶	32	
σ = 0 to 4000 psi																	
250	-----	1.20 × 10 ⁶	0.98-1.34 × 10 ⁶	44		1.26 × 10 ⁶	1.06-1.39 × 10 ⁶	37		1.18 × 10 ⁶	1.02-1.37 × 10 ⁶	30		0.49 × 10 ⁶	0.38-0.65 × 10 ⁶	19	
250	Asbestos-covered trays	1.07	.84-1.32	39		1.27	1.19-1.39	37		----	----	--		.66	.55-.86	25	
σ = 0 to 2500 psi																	
300	-----	0.78 × 10 ⁶	0.74-0.81 × 10 ⁶	28		0.80 × 10 ⁶	0.74-0.89 × 10 ⁶	23		b _{1.13} × 10 ⁶	b _{0.56-1.39} × 10 ⁶	b ₂₉		----	----	--	
300	Asbestos-covered trays	.68	.61-.73	25		.78	.66-.90	23		1.44	1.29-1.55	37		----	----	--	
375	Asbestos-covered trays	-----	-----	---		-----	-----	---		-----	-----	---		----	----	--	
Modulus of elasticity of specimens at a temperature T after 200-hr heating at the temperature T ^d																	
σ = 0 to 5000 psi																	
150	-----	2.02 × 10 ⁶	1.87-2.19 × 10 ⁶	73		2.72 × 10 ⁶	2.64-2.84 × 10 ⁶	80		3.13 × 10 ⁶	2.96-3.32 × 10 ⁶	81		0.82 × 10 ⁶	0.64-0.96 × 10 ⁶	31	
σ = 0 to 10,000 psi																	
150	-----	1.97 × 10 ⁶	1.83-2.10 × 10 ⁶	72		2.65 × 10 ⁶	2.58-2.74 × 10 ⁶	79		2.93 × 10 ⁶	2.72-3.24 × 10 ⁶	76		0.78 × 10 ⁶	0.56-0.95 × 10 ⁶	30	
σ = 0 to 4000 psi																	
200	-----	1.03 × 10 ⁶	0.97-1.08 × 10 ⁶	37		2.02 × 10 ⁶	1.93-2.15 × 10 ⁶	60		2.83 × 10 ⁶	2.76-2.93 × 10 ⁶	73		0.44 × 10 ⁶	0.40-0.47 × 10 ⁶	17	
σ = 0 to 1500 psi																	
250	-----	0.91 × 10 ⁶	0.42-1.21 × 10 ⁶	33		1.82 × 10 ⁶	1.65-2.02 × 10 ⁶	53		1.82 × 10 ⁶	1.48-2.09 × 10 ⁶	47		----	----	--	
325	-----	-----	-----	---		-----	-----	---		-----	-----	---		----	----	--	

^aReferred to control mean secant modulus for corresponding stress range; in cases where the least-stress range, 0 to 91 psi, for the control specimens is higher than the stress range for the heated specimens, the stress-deflection graph was linear within the experimental accuracy below the stress σ_1 .

^dUnless otherwise noted, all specimens which were heated for 200 hr were heated in open wide-mouthed bottles placed in a circulating-air oven.

^eNormal tray is an aluminum tray. Asbestos-covered tray is same aluminum tray covered with asbestos paper about 1/16 in. thick. Unless otherwise noted, all tests at elevated temperature were made with normal tray.

^bSpecimens not randomly selected but taken from at least three sheets.

^{1A}A muffle furnace controlled at $325^\circ \pm 10^\circ$ C was used for conditioning these specimens.

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TABLE 3.- SECANT MODULUS OF ELASTICITY FOR SPECIMENS OF GLASS-FABRIC LAMINATES TESTED UNDER VARIOUS CONDITIONS OF TEMPERATURE AND HEATING - Continued

Temperature of heating (°C)	Special conditions	Acrylic-resin laminate C			Silicone-resin laminate B			Phenolic-resin laminate H			Melamine-resin laminate J		
		Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)
Modulus of elasticity of control specimens at 25° C after at least 7 days at 25° C and 50-percent relative humidity													
σ = 0 to 2700 psi													
None	A ^b	1.40 × 10 ⁶	1.35-1.54 × 10 ⁶	---	2.11 × 10 ⁶	1.98-2.21 × 10 ⁶	--	2.55 × 10 ⁶	2.19-2.84 × 10 ⁶	---	3.31 × 10 ⁶	3.25-3.38 × 10 ⁶	--
Do----	B ^b	1.41	1.33-1.49	---	2.04	1.97-2.12	---	2.64	2.59-3.01	---	3.26	3.14-3.39	--
Do----	Control mean	1.40	---	---	2.08	---	---	2.59	---	---	3.29	---	---
σ = 0 to 10,000 psi													
Do----	A ^b	1.39 × 10 ⁶	1.35-1.54 × 10 ⁶	---	2.10 × 10 ⁶	1.98-2.20 × 10 ⁶	--	2.55 × 10 ⁶	2.19-2.84 × 10 ⁶	---	3.29 × 10 ⁶	3.21-3.37 × 10 ⁶	--
Do----	B ^b	1.41	1.33-1.49	---	2.03	1.96-2.12	---	2.62	2.59-3.00	---	3.21	3.13-3.31	--
Do----	Control mean	1.40	---	---	2.07	---	---	2.58	---	---	3.25	---	---
σ = 0 to 20,000 psi													
Do----	A ^b	1.30 × 10 ⁶	1.25-1.34 × 10 ⁶	---	2.08 × 10 ⁶	1.98-2.16 × 10 ⁶	---	2.55 × 10 ⁶	2.19-2.84 × 10 ⁶	---	3.26 × 10 ⁶	3.18-3.32 × 10 ⁶	--
Do----	B ^b	1.32	1.26-1.44	---	2.02	1.94-2.11	---	2.59	2.25-2.99	---	3.20	3.13-3.29	--
Do----	Control mean	1.31	---	---	2.05	---	---	2.56	---	---	3.23	---	---
σ = 0 to 30,000 psi													
Do----	A ^b	---	---	---	1.98 × 10 ⁶	1.87-2.07 × 10 ⁶	---	2.51 × 10 ⁶	2.16-2.82 × 10 ⁶	---	3.15 × 10 ⁶	3.09-3.23 × 10 ⁶	---
Do----	B ^b	---	---	---	1.92	1.84-2.01	---	2.56	2.24-2.94	---	3.12	3.07-3.21	---
Do----	Control mean	---	---	---	1.95	---	---	2.53	---	---	3.14	---	---
σ = 0 to 30,000 psi ^d													
Do----	A ^b	---	---	---	---	---	---	2.48 × 10 ⁶	2.13-2.78 × 10 ⁶	---	---	---	---
Do----	B ^b	---	---	---	---	---	---	2.53	2.19-2.94	---	---	---	---
Do----	Control mean	---	---	---	---	---	---	2.50	---	---	---	---	---
Modulus of elasticity of specimens at 25° C after 200-hr heating ^d (reconditioned 7 days at 25° C and 50-percent relative humidity, unless otherwise noted)													
σ = 0 to 10,000 psi													
150	Not reconditioned ^e	1.37 × 10 ⁶	1.32-1.49 × 10 ⁶	98	2.03 × 10 ⁶	1.97-2.06 × 10 ⁶	99	2.50 × 10 ⁶	2.30-2.71 × 10 ⁶	98	2.42 × 10 ⁶	2.35-2.47 × 10 ⁶	74
150	---	1.43	1.26-1.60	106	1.98	1.87-2.16	97	2.42	2.33-2.61	95	2.44	2.37-2.51	75
σ = 0 to 20,000 psi													
150	Not reconditioned ^e	1.34 × 10 ⁶	1.29-1.45 × 10 ⁶	102	1.91 × 10 ⁶	1.87-1.96 × 10 ⁶	98	2.44 × 10 ⁶	2.27-2.70 × 10 ⁶	98	2.41 × 10 ⁶	2.34-2.49 × 10 ⁶	68
150	---	1.43	1.22-1.53	109	1.88	1.77-2.06	96	2.33	2.24-2.47	95	1.97	1.89-2.04	63
σ = 0 to 15,000 psi													
200	Not heated in bottles ^f	---	---	---	---	---	---	1.72 × 10 ⁶	1.59-1.88 × 10 ⁶	66	2.06 × 10 ⁶	2.00-2.10 × 10 ⁶	63
200	---	---	---	---	---	---	---	1.28	1.06-1.57	49	2.01	1.93-2.11	62
σ = 0 to 10,000 psi													
250	---	---	---	---	1.84 × 10 ⁶	1.78-1.87 × 10 ⁶	94	(k)	(k)	---	1.89 × 10 ⁶	1.80-1.95 × 10 ⁶	57

^aReferred to control mean secant modulus for corresponding stress range; in cases where the least-stress range, 0 to 0.1 psi, for the control specimens is higher than the stress range for the heated specimens, the stress-deflection graph was linear within the experimental accuracy below the stress σ_1 .

^bA, first set of controls (Oct. 1948); B, second set of controls (April 1949).

^cUnless otherwise noted, all specimens which were heated for 200 hr were heated in open wide-mouthed bottles placed in a circulating-air oven.

^dAfter removal from oven, specimens kept over calcium chloride for 24-hr prior to testing.

^eSpecimens laid on asbestos board on oven shelf.

^fFour specimens.

^gLoad-deflection records were too small to permit determination of modulus.

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TABLE 3.- SECANT MODULUS OF ELASTICITY FOR SPECIMENS OF GLASS-FABRIC LAMINATES TESTED UNDER VARIOUS CONDITIONS OF TEMPERATURE AND HEATING - Concluded

Temperature of heating (°C)	Special conditions	Acrylic-resin laminate C			Silicone-resin laminate B			Phenolic-resin laminate H			Melamine-resin laminate J		
		Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)	Mean (psi)	Range (psi)	Percent of control mean (a)
150	-----	Modulus of elasticity of specimens at a temperature T after 0.5-hr heating at temperature T											
		$\sigma = 0$ to 1000 psi											
		0.29×10^6	$0.26-0.32 \times 10^6$	21	1.39×10^6	$1.31-1.48 \times 10^6$	67	2.44×10^6	$2.30-2.54 \times 10^6$	95	2.33×10^6	$2.19-2.42 \times 10^6$	72
150	-----	$\sigma = 0$ to 2000 psi											
		0.26×10^6	$0.25-0.29 \times 10^6$	19	1.26×10^6	$1.19-1.33 \times 10^6$	61	2.40×10^6	$2.27-2.50 \times 10^6$	96	2.05×10^6	$1.88-2.16 \times 10^6$	65
		$\sigma = 0$ to 20,000 psi											
200	-----	$\sigma = 0$ to 20,000 psi											
		$\sigma = 0$ to 15,000 psi											
		$\sigma = 0$ to 15,000 psi											
250	Asbestos-covered trays ¹	$\sigma = 0$ to 7500 psi											
		1.36×10^6	$1.27-1.41 \times 10^6$	66	1.52×10^6	$1.42-1.79 \times 10^6$	59	1.68×10^6	$1.60-1.73 \times 10^6$	52			
		-----	-----	--	1.77	$1.52-1.90$	69	1.73	$1.68-1.79$	53			
300	Asbestos-covered trays ¹	$\sigma = 0$ to 7500 psi											
		1.35×10^6	$1.30-1.44 \times 10^6$	65	1.69×10^6	$1.27-2.11 \times 10^6$	66	1.60×10^6	$1.56-1.65 \times 10^6$	49			
		1.39	$1.32-1.52$	67	1.99	$1.36-1.79$	62	1.72	$1.65-1.75$	53			
375	Asbestos-covered trays ¹	$\sigma = 0$ to 7500 psi											
		1.25×10^6	$1.11-1.41 \times 10^6$	60	0.99×10^6	$0.84-1.08 \times 10^6$	38	(k)	(k)	--			
		-----	-----	--									
150	-----	Modulus of elasticity of specimens at a temperature T after 200-hr heating at temperature T ¹											
		$\sigma = 0$ to 2000 psi											
		0.32×10^6	$0.16-0.50 \times 10^6$	23	1.54×10^6	$1.42-1.65 \times 10^6$	74	2.46×10^6	$2.34-2.62 \times 10^6$	96	2.41×10^6	$2.28-2.49 \times 10^6$	73
150	-----	$\sigma = 0$ to 2700 psi											
		0.30×10^6	$0.13-0.48 \times 10^6$	21	1.51×10^6	$1.35-1.63 \times 10^6$	74	2.37×10^6	$2.20-2.60 \times 10^6$	95	2.23×10^6	$2.11-2.36 \times 10^6$	69
		$\sigma = 0$ to 7500 psi											
200	-----	$\sigma = 0$ to 7500 psi											
		$\sigma = 0$ to 15,000 psi											
		$\sigma = 0$ to 15,000 psi											
250	-----	$\sigma = 0$ to 7500 psi											
		1.66×10^6	$1.62-1.73 \times 10^6$	80	(k)	(k)	--	1.21×10^6	$1.15-1.28 \times 10^6$	37			
		$\sigma = 0$ to 1000 psi											
1325	-----	$\sigma = 0$ to 1000 psi											
		1.61×10^6	$1.50-1.76 \times 10^6$	78	-----	-----	--	1.04×10^6	$0.92-0.75 \times 10^6$	m19			
		$\sigma = 0$ to 1000 psi											

^aReferred to control mean secant modulus for corresponding stress range; in cases where the least-stress range, 0 to 0.1 psi, for the control specimens is higher than the stress range for the heated specimens, the stress-deflection graph was linear within the experimental accuracy below the stress σ_1 .

^bNormal tray is an aluminum tray. Asbestos-covered tray is same aluminum tray covered with asbestos paper about 1/16 in. thick. Unless otherwise noted, all tests at elevated temperature were made with normal tray.

^cSpecimens not randomly selected but taken from at least three sheets.

^dA muffle furnace controlled at $3250 \pm 10^\circ \text{C}$ was used for conditioning these specimens.

^eFour specimens.

^fLoad-deflection records were too small to permit determination of modulus.

^gTwo specimens.

^hReferred to control value for 0 to 10,000 psi, although the control stress-deflection graph was slightly curved below 10,000 psi.



TABLE 4.- LOSS OF WEIGHT AND LOSS OF RESIN BY GLASS-FABRIC LAMINATES AFTER 200-HR HEATING AT VARIOUS TEMPERATURES

[Initial weights were determined after at least 7-day conditioning at 25° C and 50-percent relative humidity; final weights were obtained after specimens had been reconditioned 7 days at 25° C and 50-percent relative humidity following the 200-hr heating. Each value is the average of results for five specimens.]

NBS sample designation	150° C		200° C		250° C		325° C	
	Weight loss (percent)	Resin loss (percent) (1)	Weight loss (percent)	Resin loss (percent) (1)	Weight loss (percent)	Resin loss (percent) (1)	Weight loss (percent)	Resin loss (percent) (1)
Unsaturated-polyester resin								
A	2.1	6.0	7.7	22.0	26.2	74.8	---	---
E	.9	2.8	4.8	15.0	25.3	79.1	---	---
F	1.2	5.0	4.6	19.2	18.1	75.4	---	---
G	2.1	5.0	10.4	24.8	31.3	74.5	---	---
Acrylic resin								
C	1.3	2.1	---	---	---	---	---	---
Silicone resin								
B	0.13	0.33	---	---	0.51	1.3	2.1	5.4
Phenolic resin								
H	2.1	4.8	8.9	20.2	34.7	79.	---	---
Melamine resin								
J	3.5	7.6	4.9	10.7	8.4	18.3	---	---

¹Calculated on basis of NBS values for resin content except for laminate B; assumption is made that total weight loss is resin.



TABLE 5.- RELATIVE HEAT RESISTANCE OF GLASS-FABRIC LAMINATES BASED ON FLEXURAL PROPERTIES

NBS sample designation	Temperature T at which flexural strength is at least 30 percent of control value for the three types of tests ¹ (°C)	Temperature T at which flexural secant modulus of elasticity is at least 50 percent of control value for the three types of test ¹	
		Temperature (°C)	Stress range. (psi)
Unsaturated-polyester resin			
A	150	150	0-6000
E	150	200	0-7500
F	140 (estimated)	200	0-4000
G	130 (estimated)	Less than 150	0-4000
Acrylic resin			
C	Much less than 150	Much less than 150	0-5000
Silicone resin			
B	325	325 (for 65 percent, estimated)	0-7500
Phenolic resin			
H	200	220 (estimated)	0-5000
Melamine resin			
J	250	220 (estimated)	0-10,000

¹Tests at 25° C after 200-hr heating at temperature T, and tests at temperature T after 0.5- or 200-hr conditioning at temperature T.



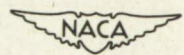
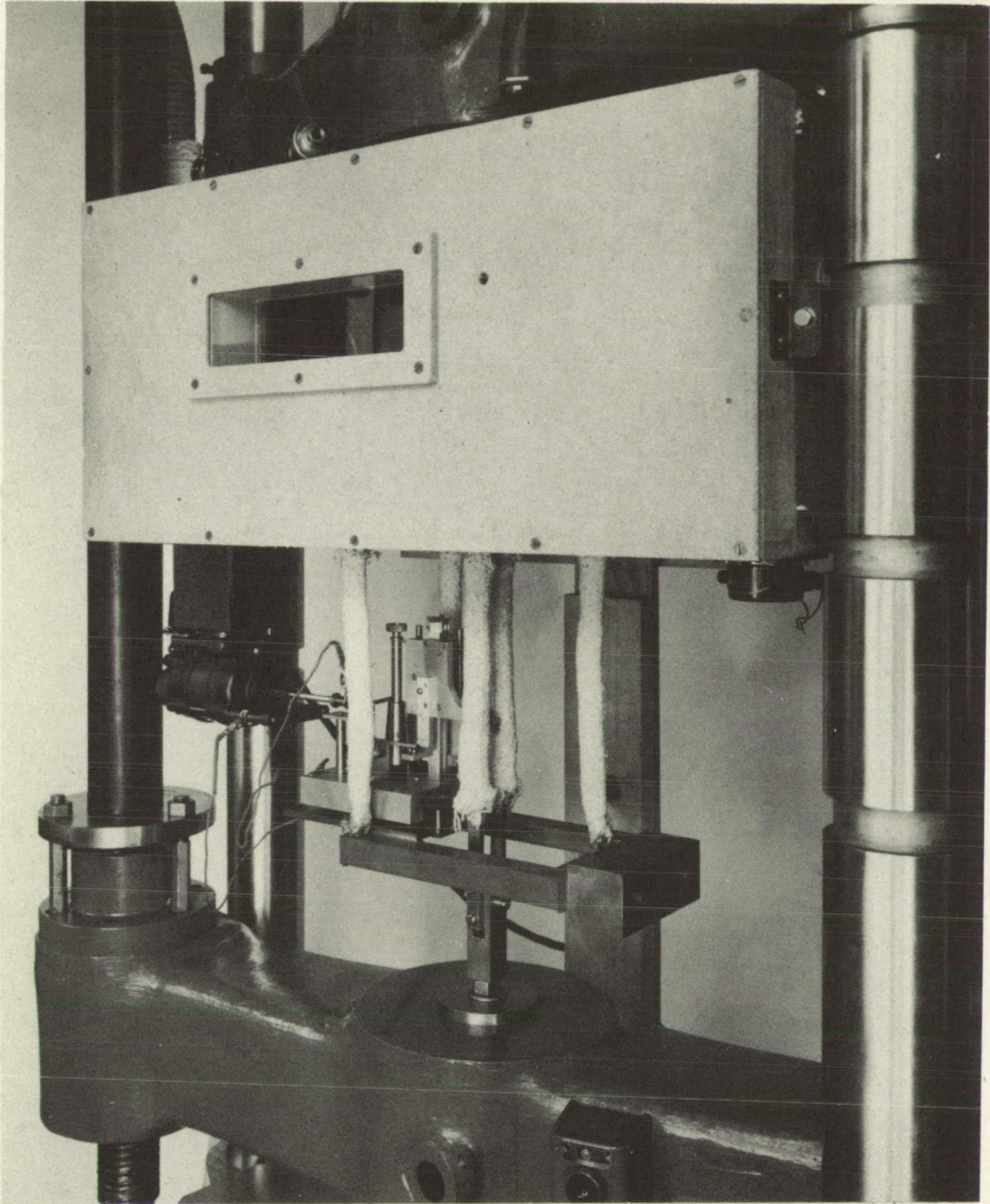


Figure 1.- Front view of flexural test enclosure in place in testing machine.
Recording deflectometer and selsyn motor are at the left below enclosure.

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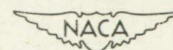
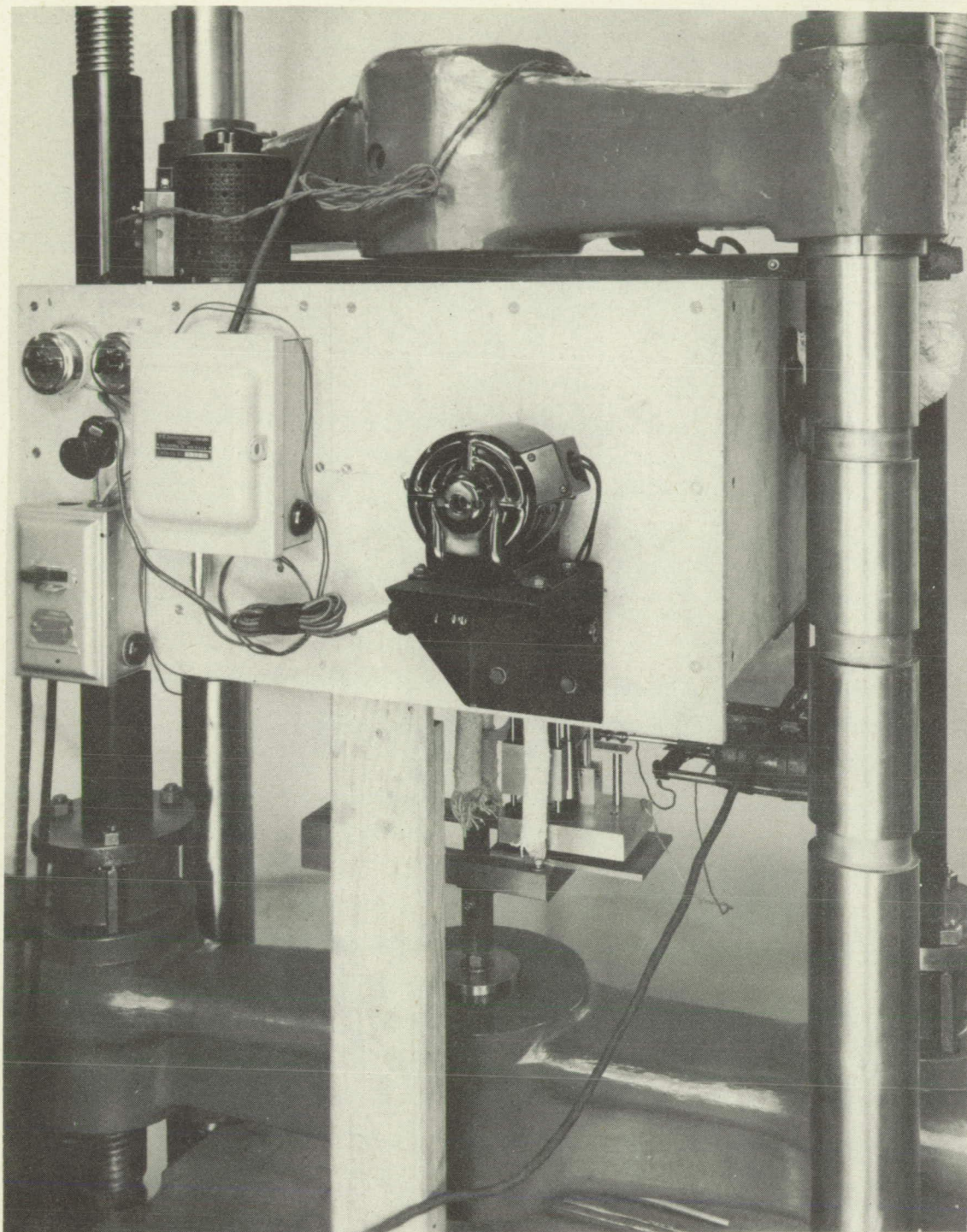


Figure 2.- Rear view of flexural test enclosure showing fan motor and various controls.

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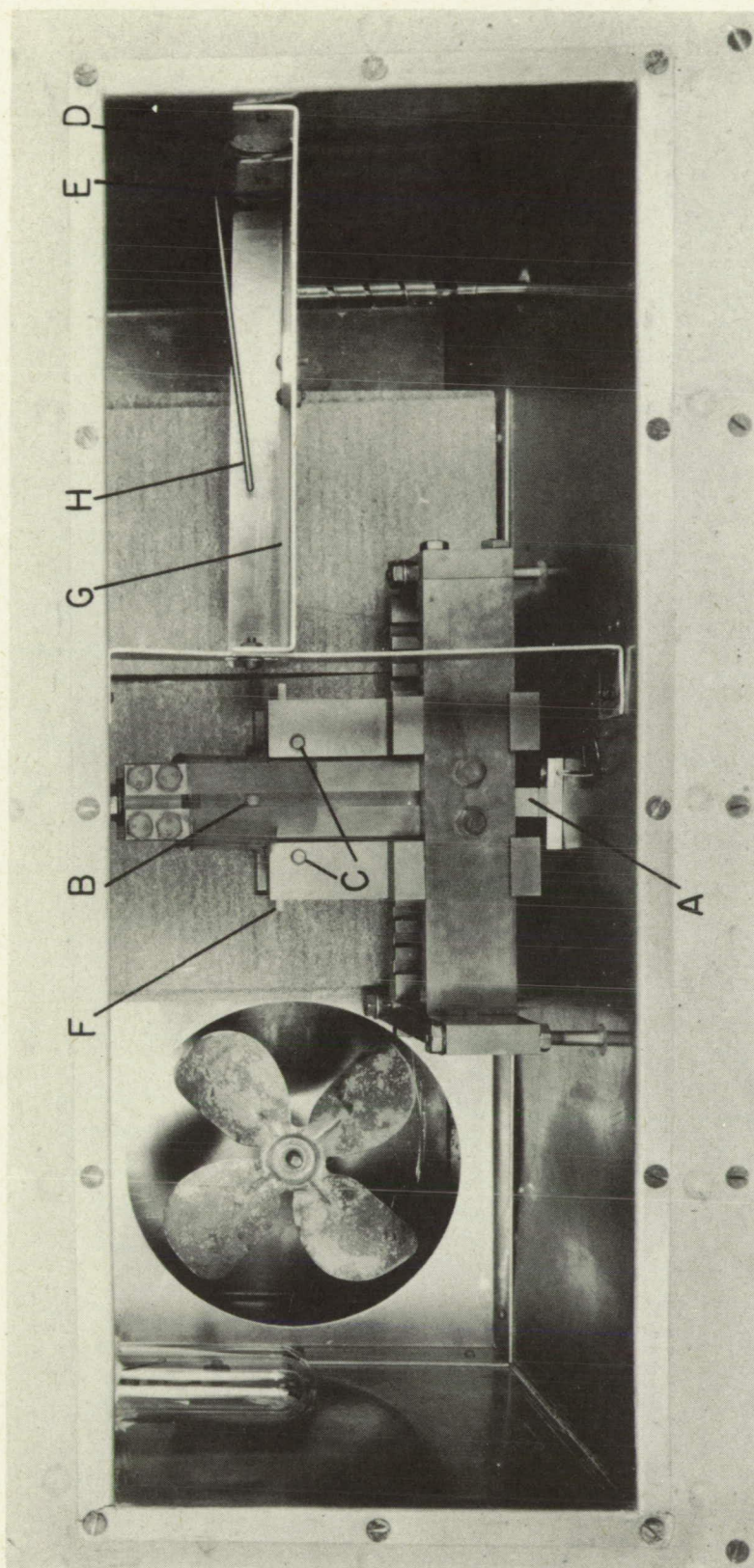


Figure 3.- Interior view of test enclosure.

- A Loading stirrup
- B Loading edge in loading stirrup
- C Contact edges in support block
- D Loading port
- E Hole for push rod
- F Specimen in jig
- G Specimen in tray
- H Stem of dial-type thermometer



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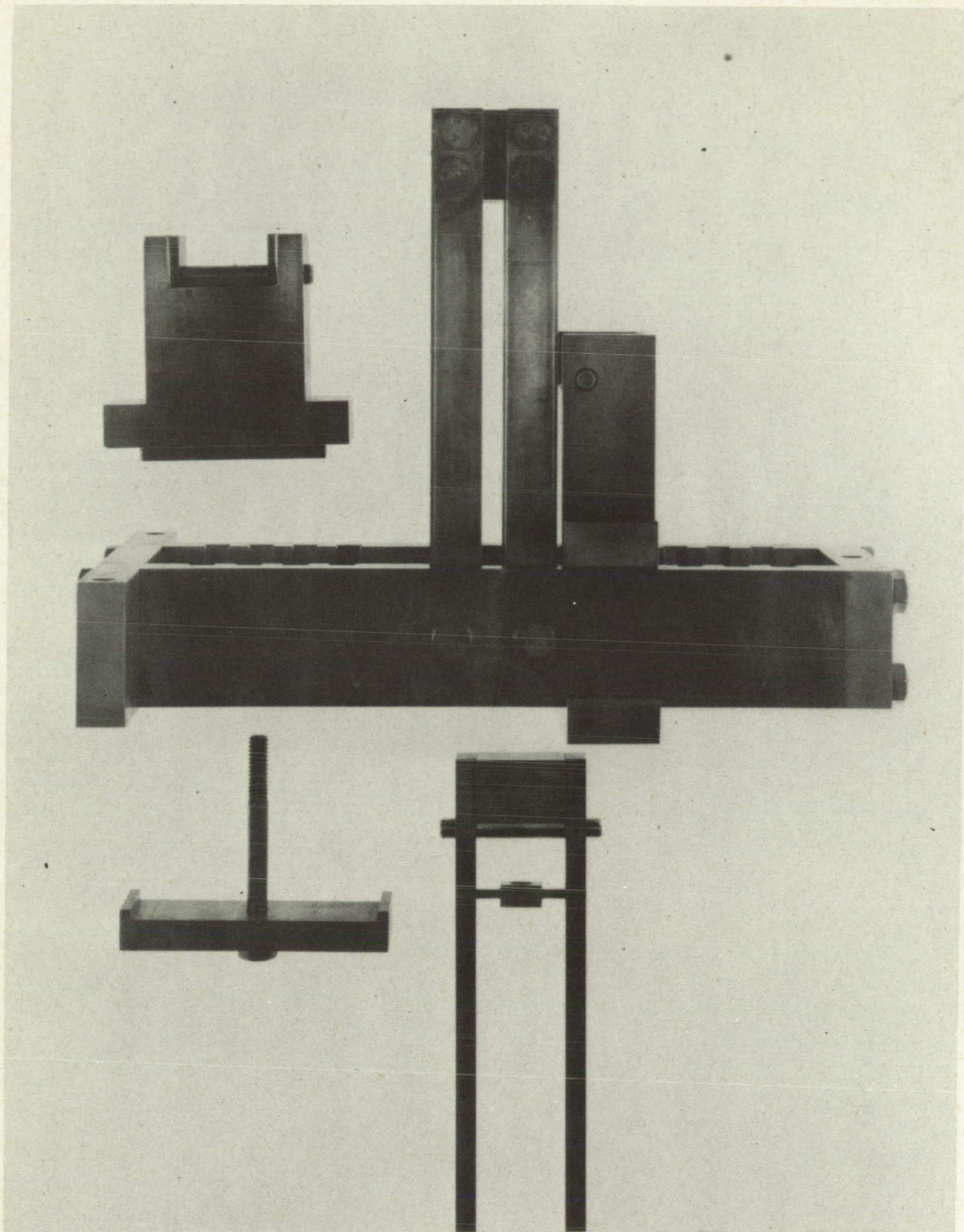


Figure 4.- Flexural jig partly disassembled; left support block and clamp and loading stirrup have been removed and rotated through 90° .

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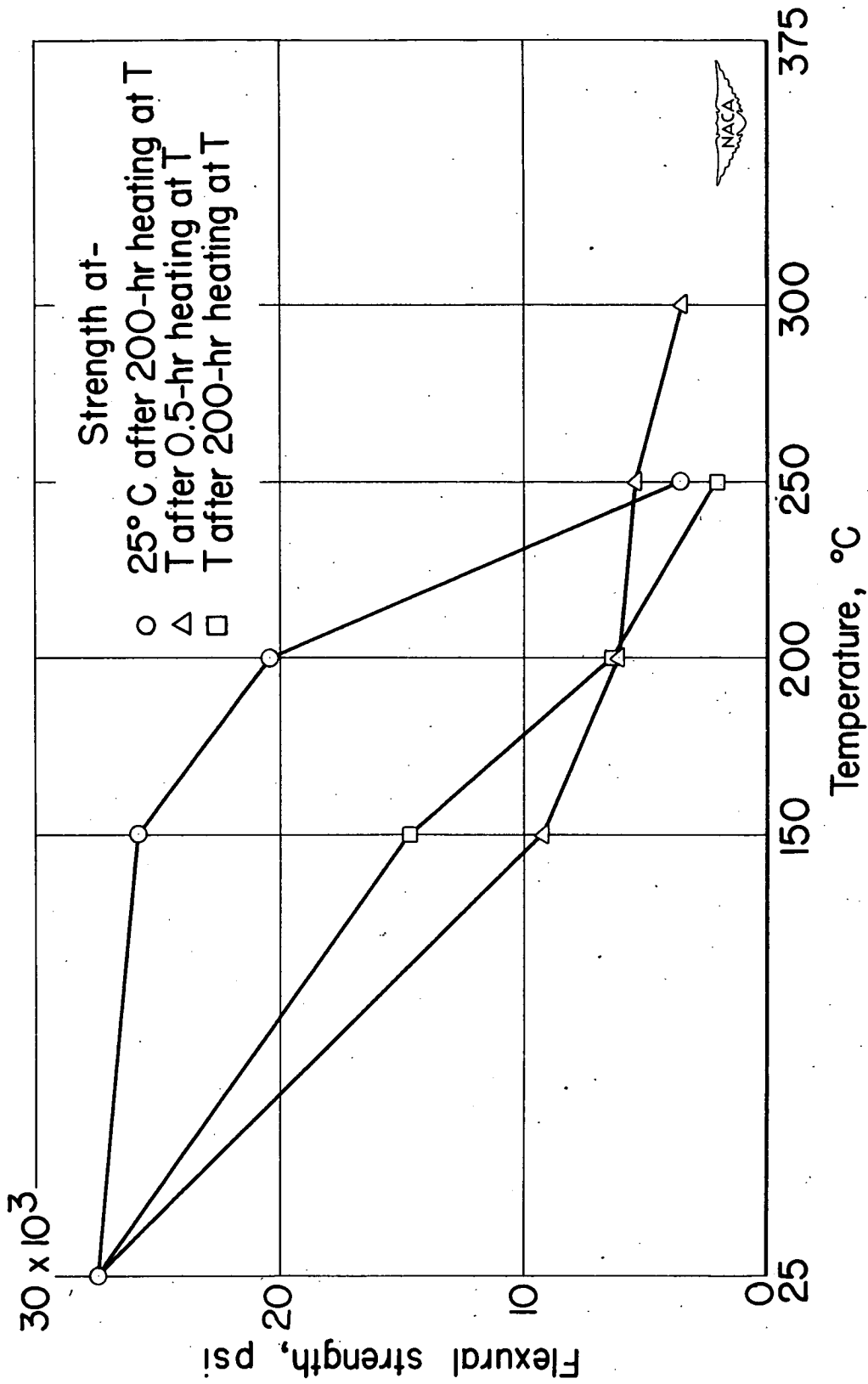


Figure 5.- Flexural strength of unsaturated-polyester laminate A for various conditions of heating and testing.

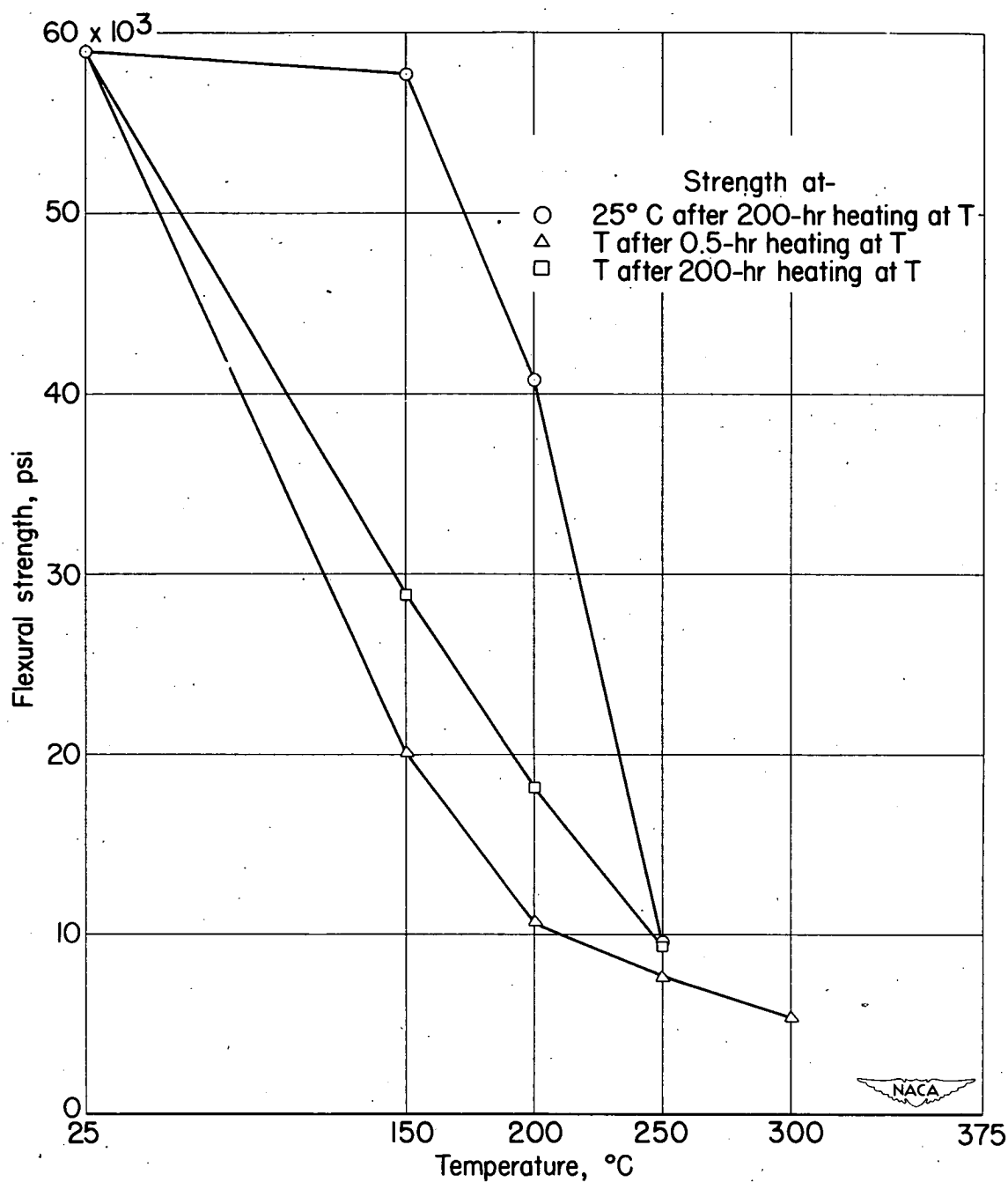


Figure 6.- Flexural strength of unsaturated-polyester laminate E for various conditions of heating and testing.

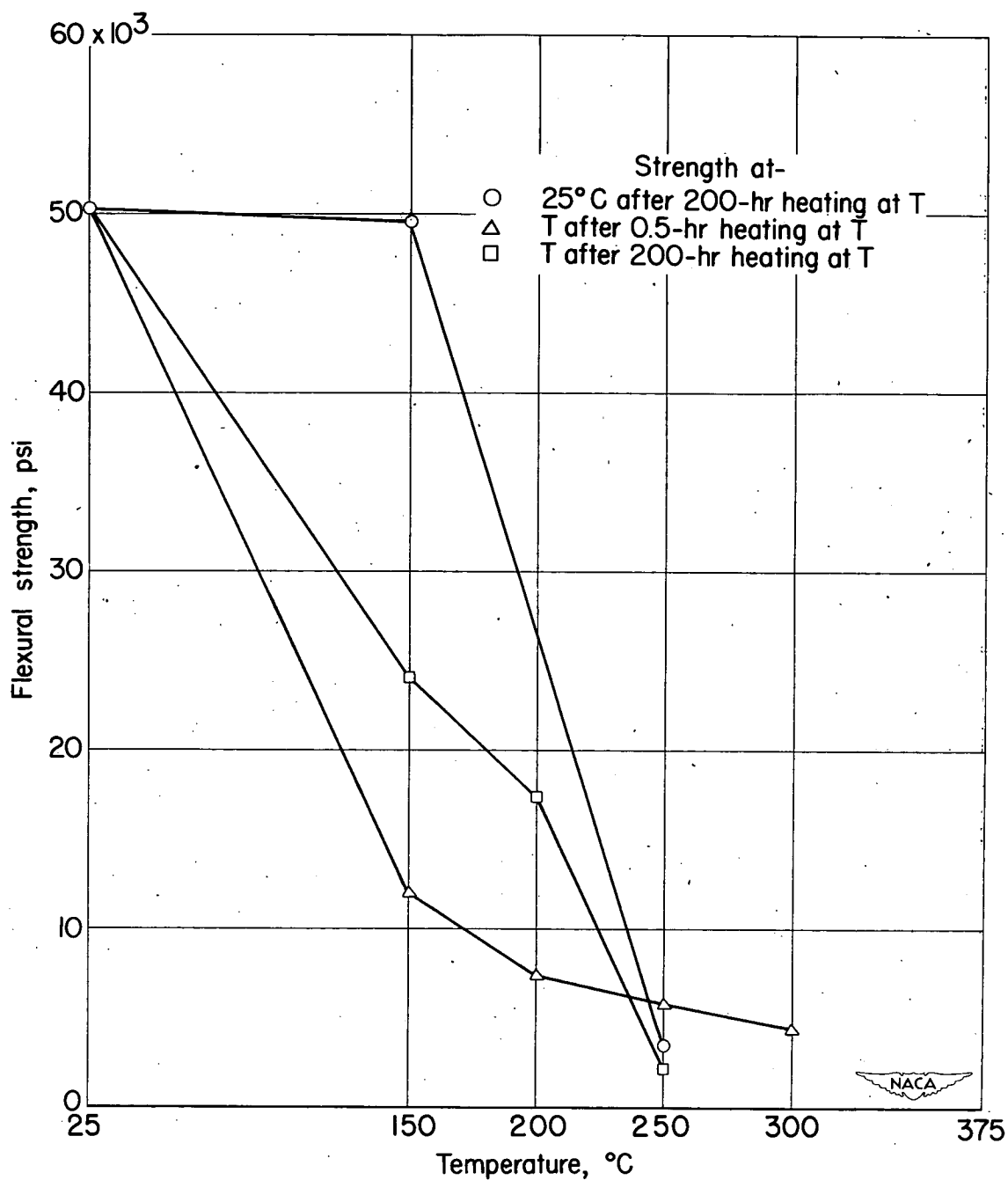


Figure 7.- Flexural strength of unsaturated-polyester laminate F for various conditions of heating and testing.

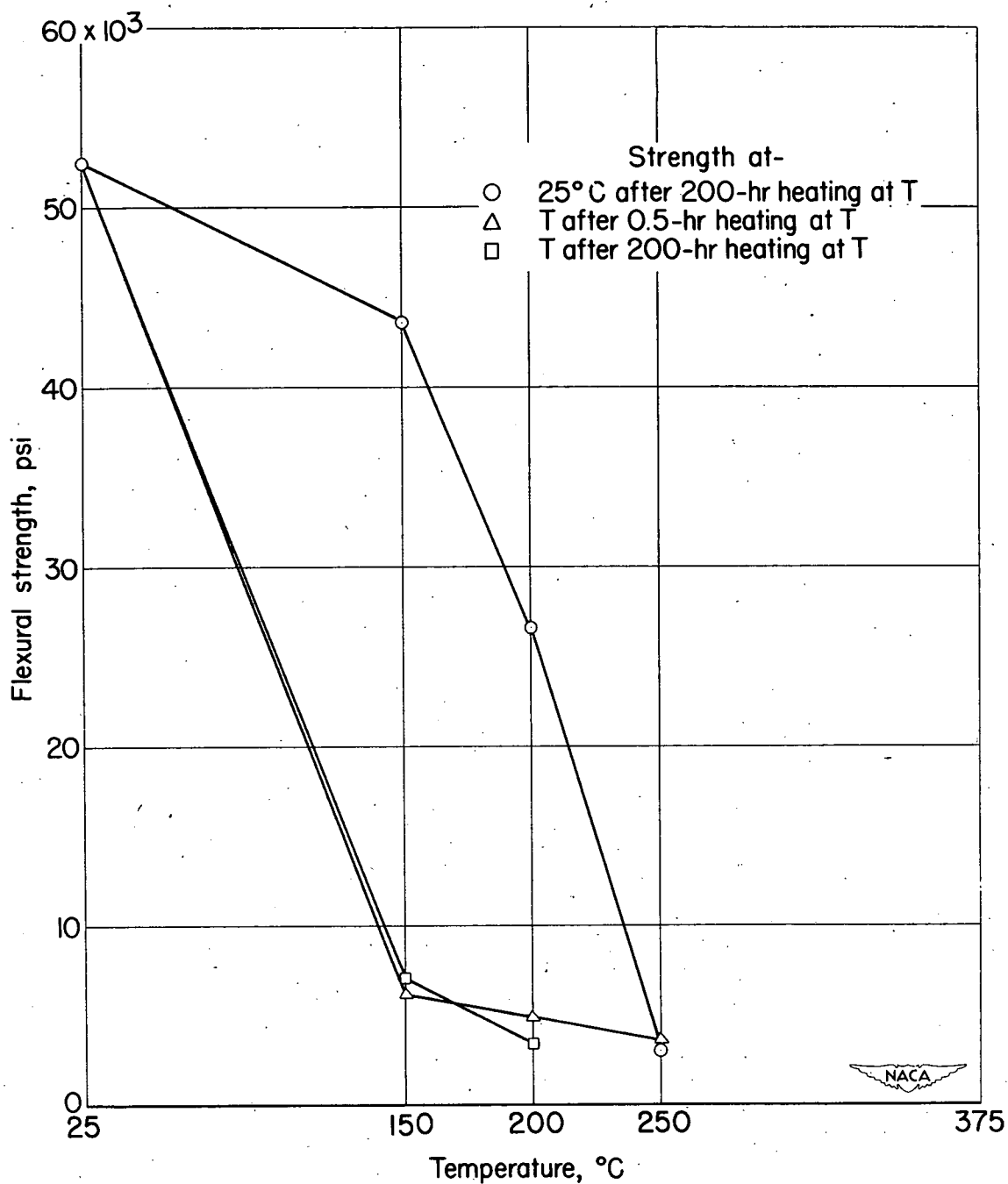


Figure 8.- Flexural strength of unsaturated-polyester laminate G for various conditions of heating and testing.

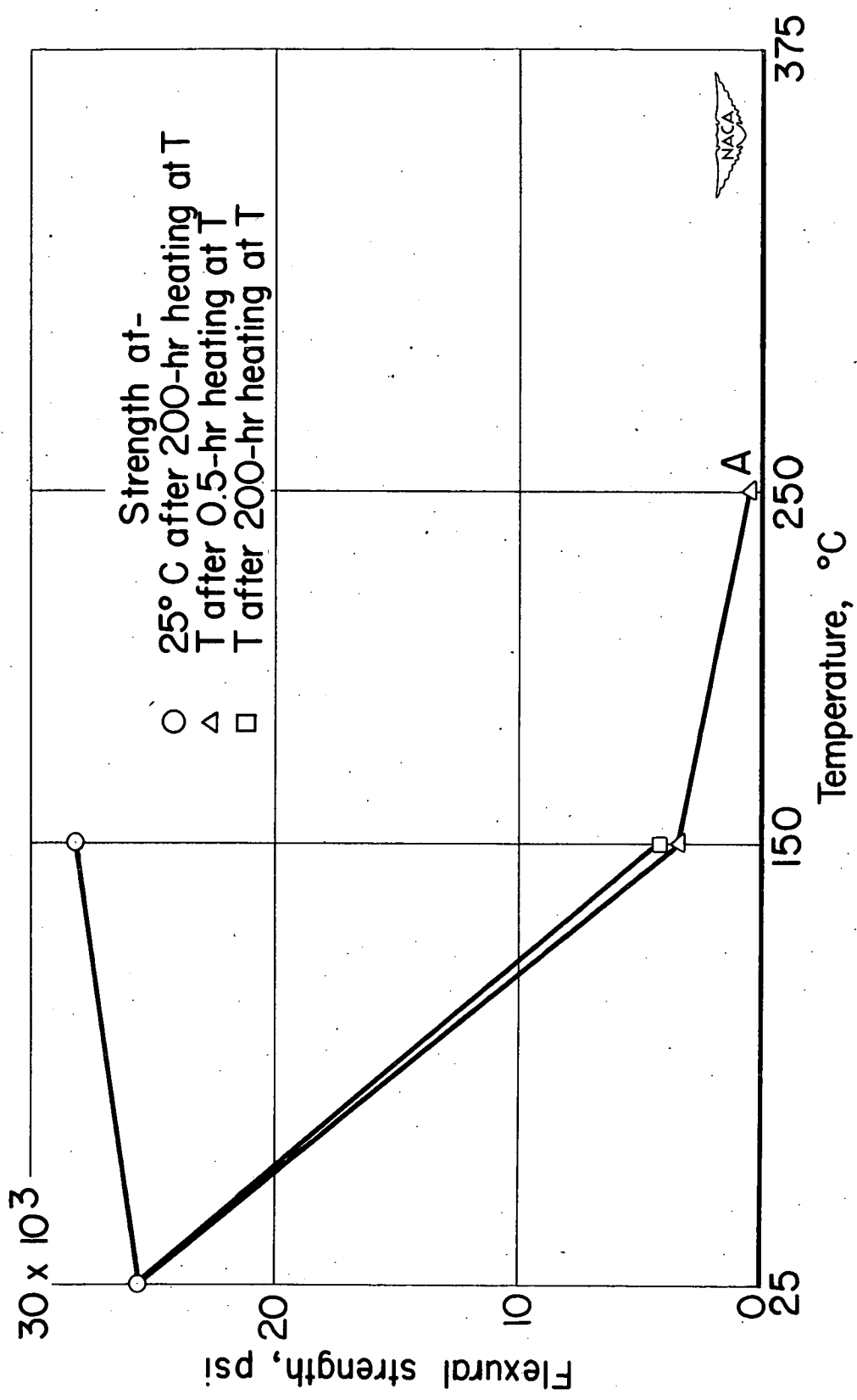


Figure 9.- Flexural strength of acrylic laminate C for various conditions of heating and testing. A, result of test in which specimen tray was covered with asbestos paper.

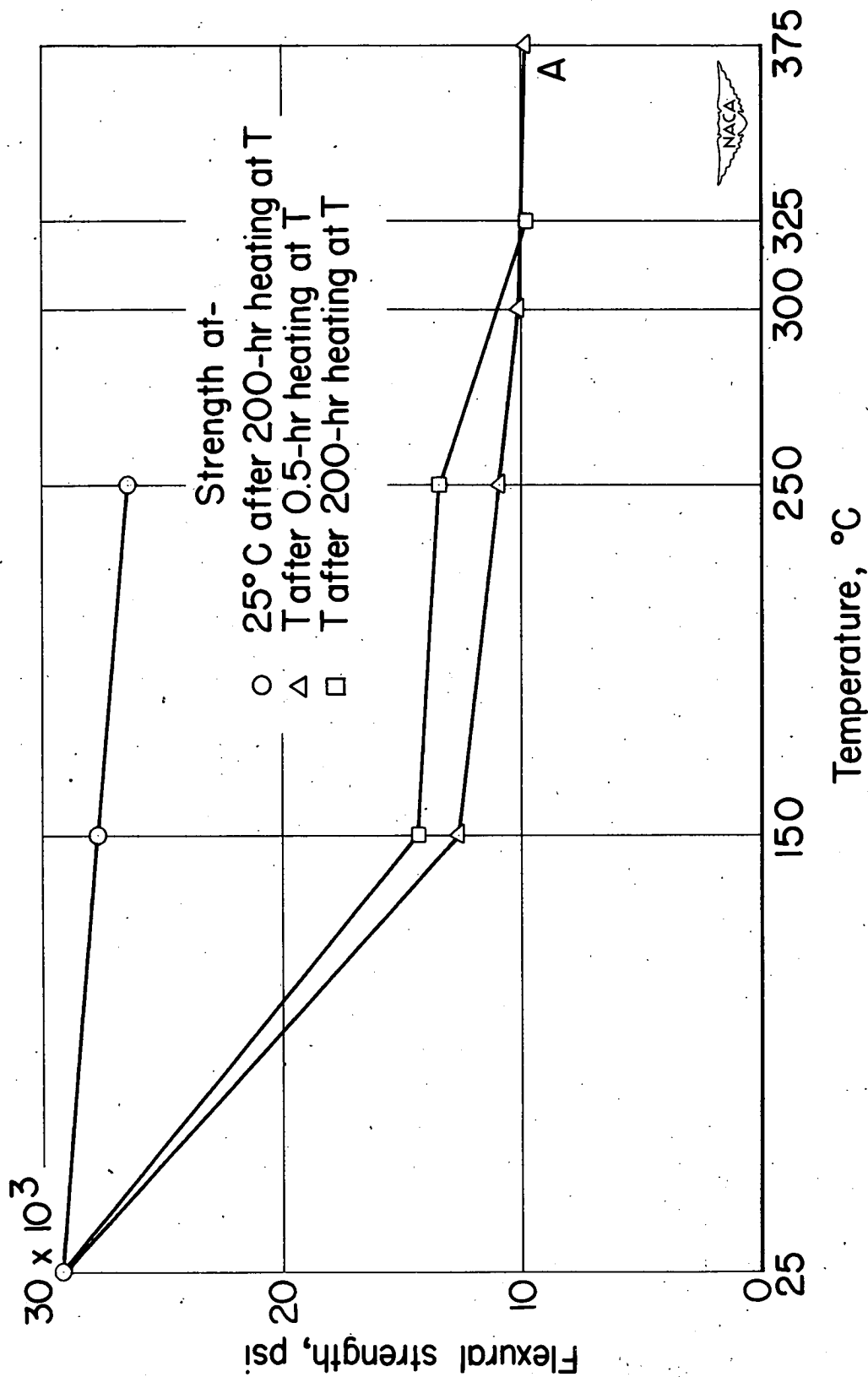


Figure 10.- Flexural strength of silicone laminate B for various conditions of heating and testing. A, result of test in which specimen tray was covered with asbestos paper.

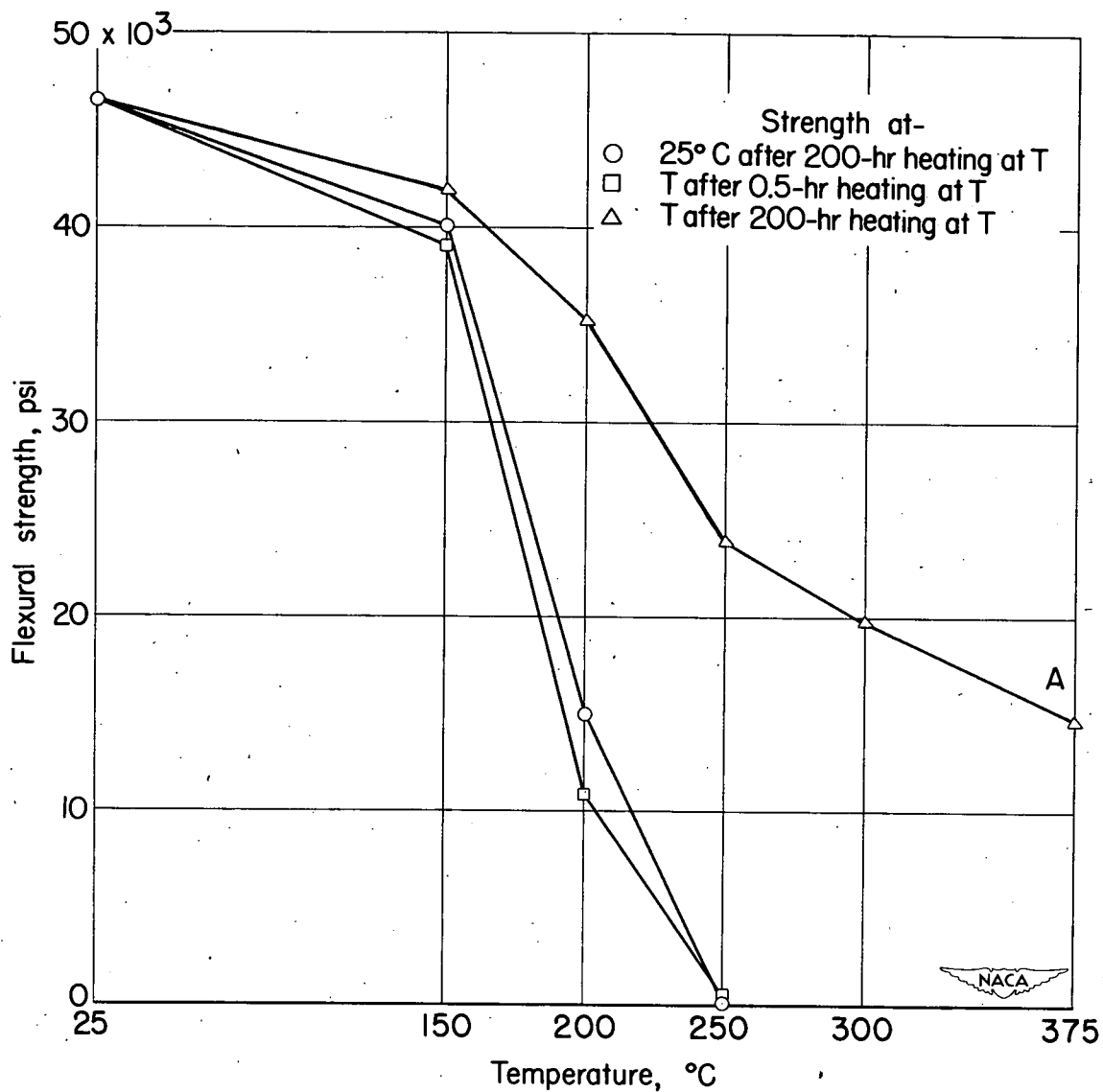


Figure 11.- Flexural strength of phenolic laminate H for various conditions of heating and testing. A, result of test in which specimen tray was covered with asbestos paper.

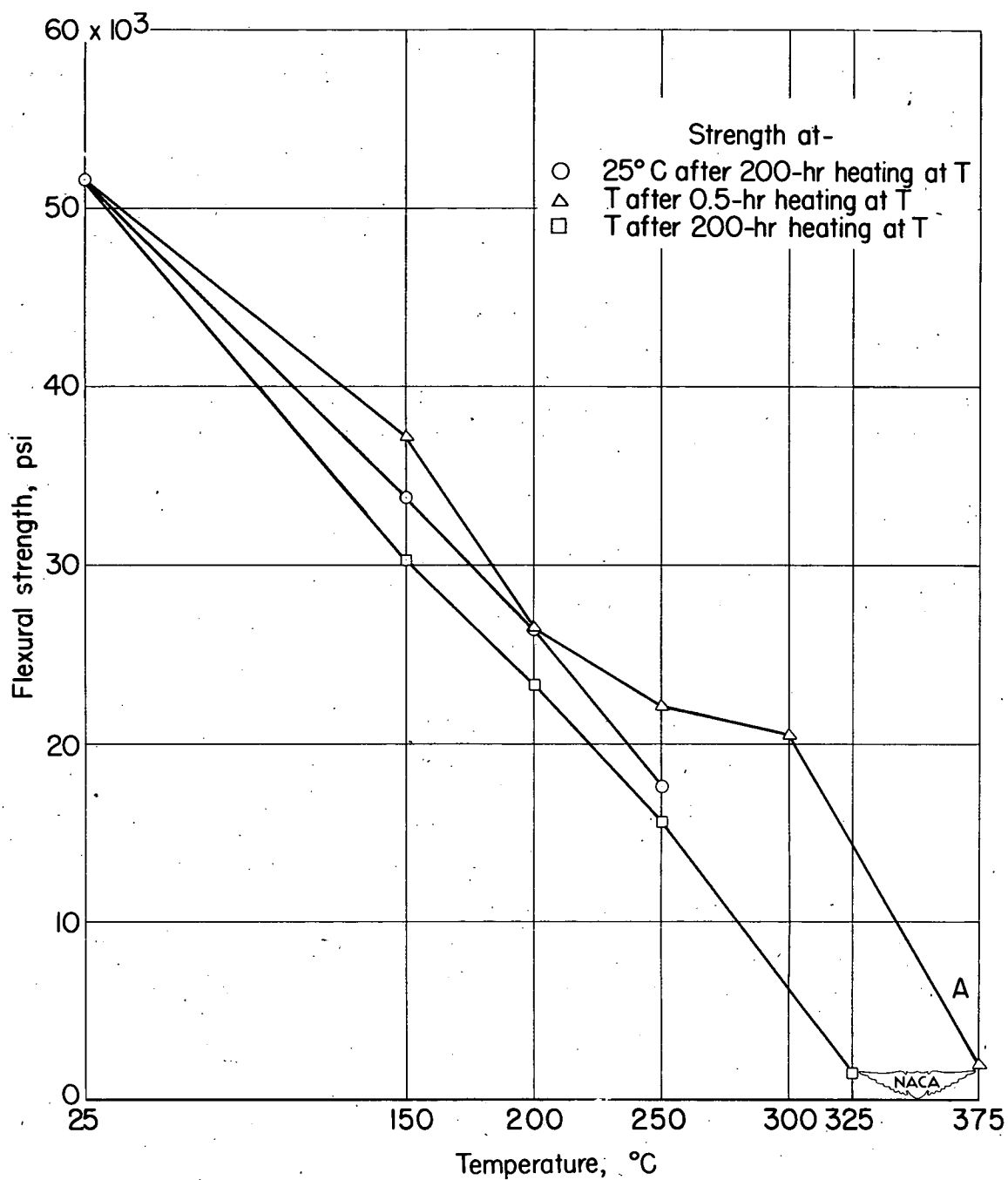


Figure 12.- Flexural strength of melamine laminate J for various conditions of heating and testing. A, result of test in which specimen tray was covered with asbestos paper.

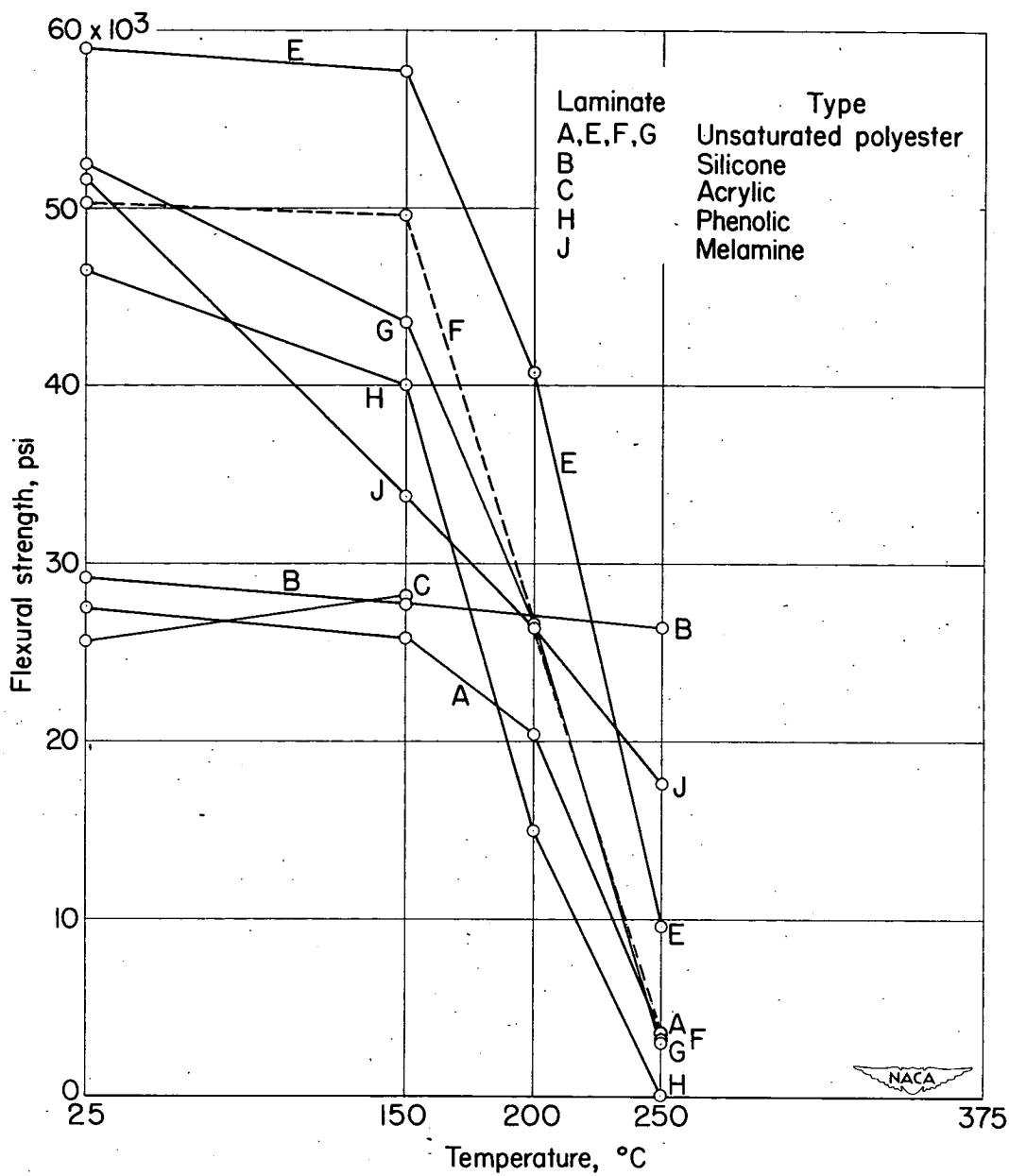


Figure 13.- Flexural strength of laminates at 25° C after 200-hour heating at various temperatures. (Data taken from figs. 5 to 12.)

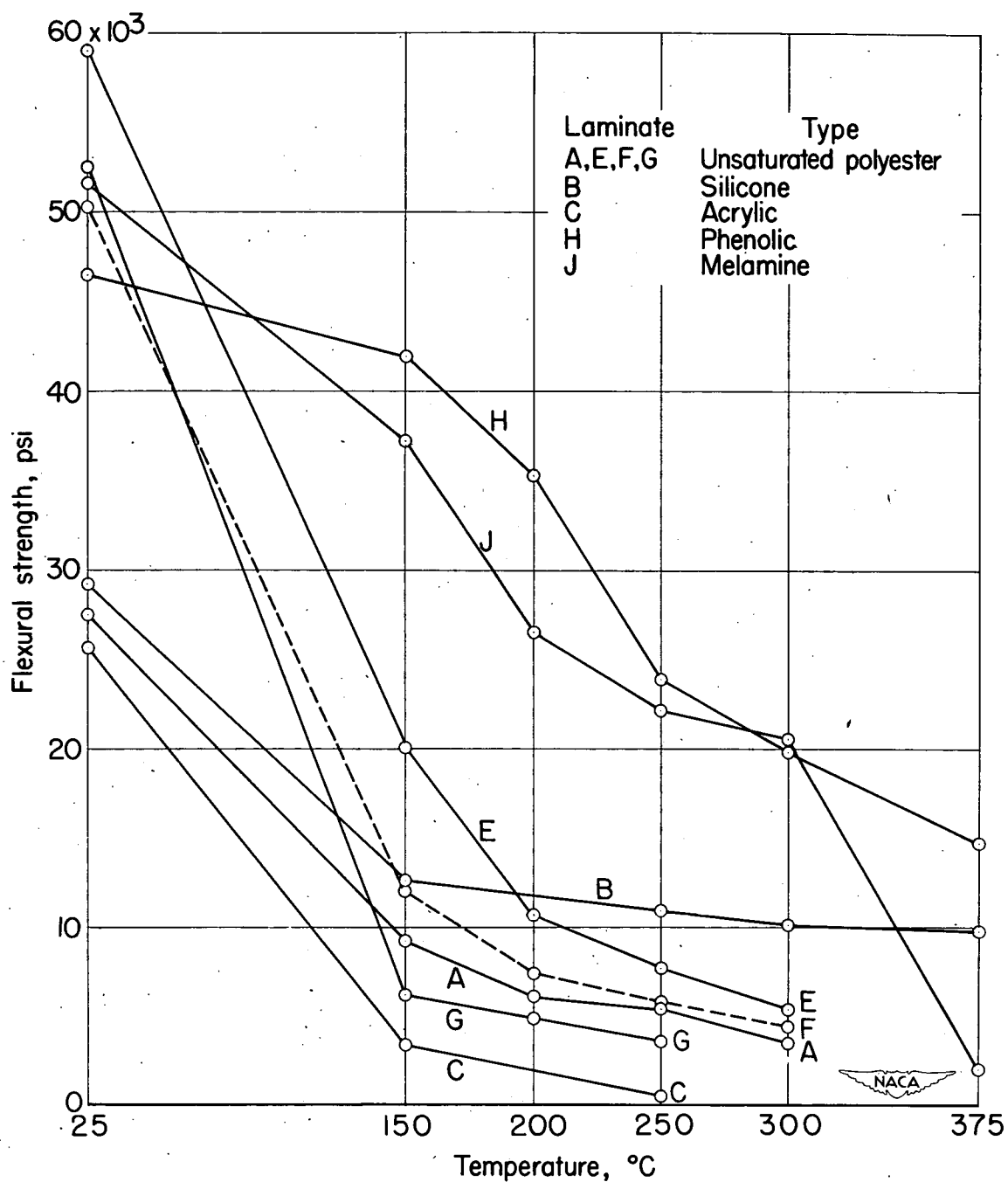


Figure 14.- Flexural strength of laminates at a temperature T after 0.5-hour heating at T . (Data taken from figs. 5 to 12.)

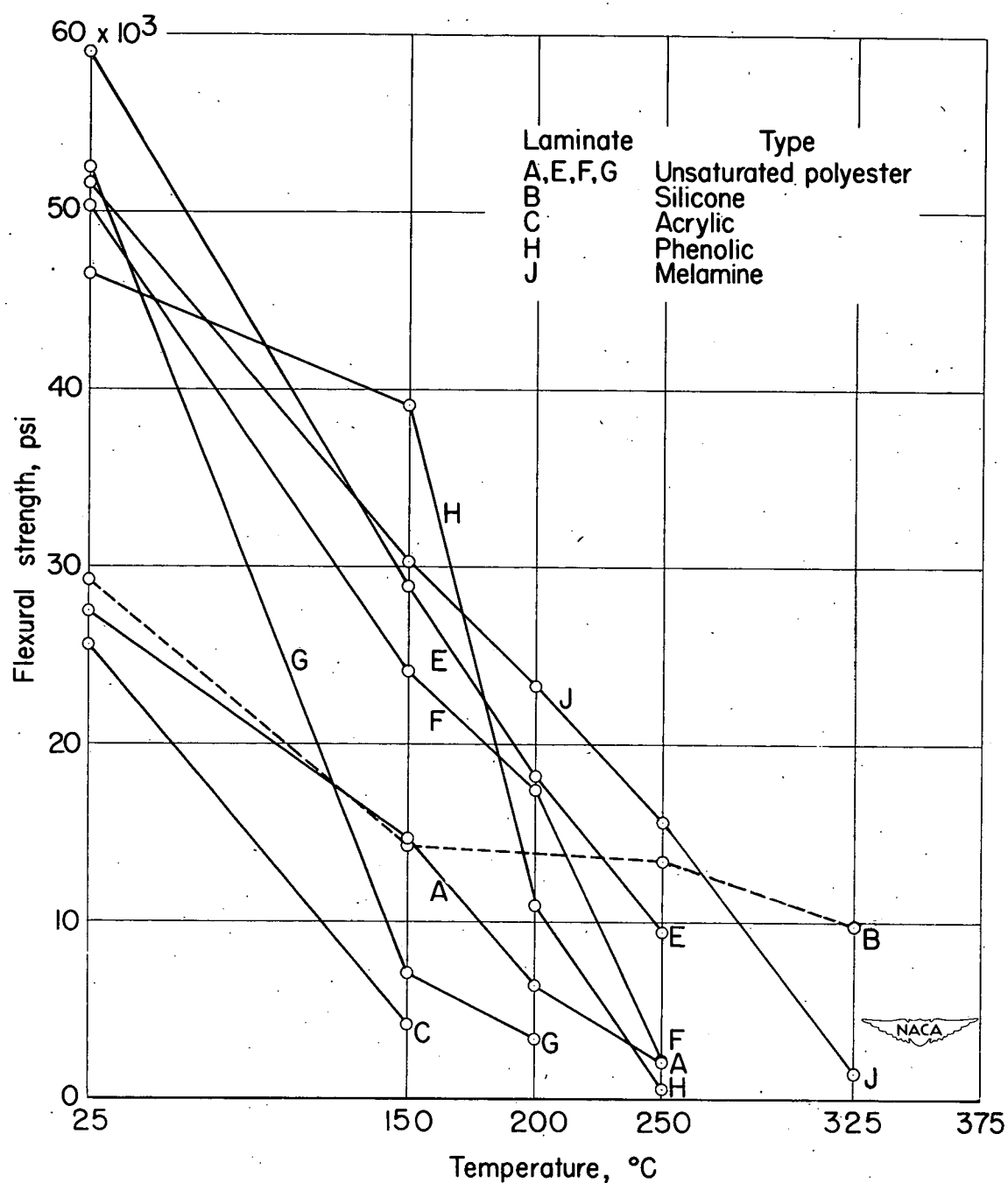


Figure 15.- Flexural strength of laminates at a temperature T after 200-hour heating at T . (Data taken from figs. 5 to 12.)

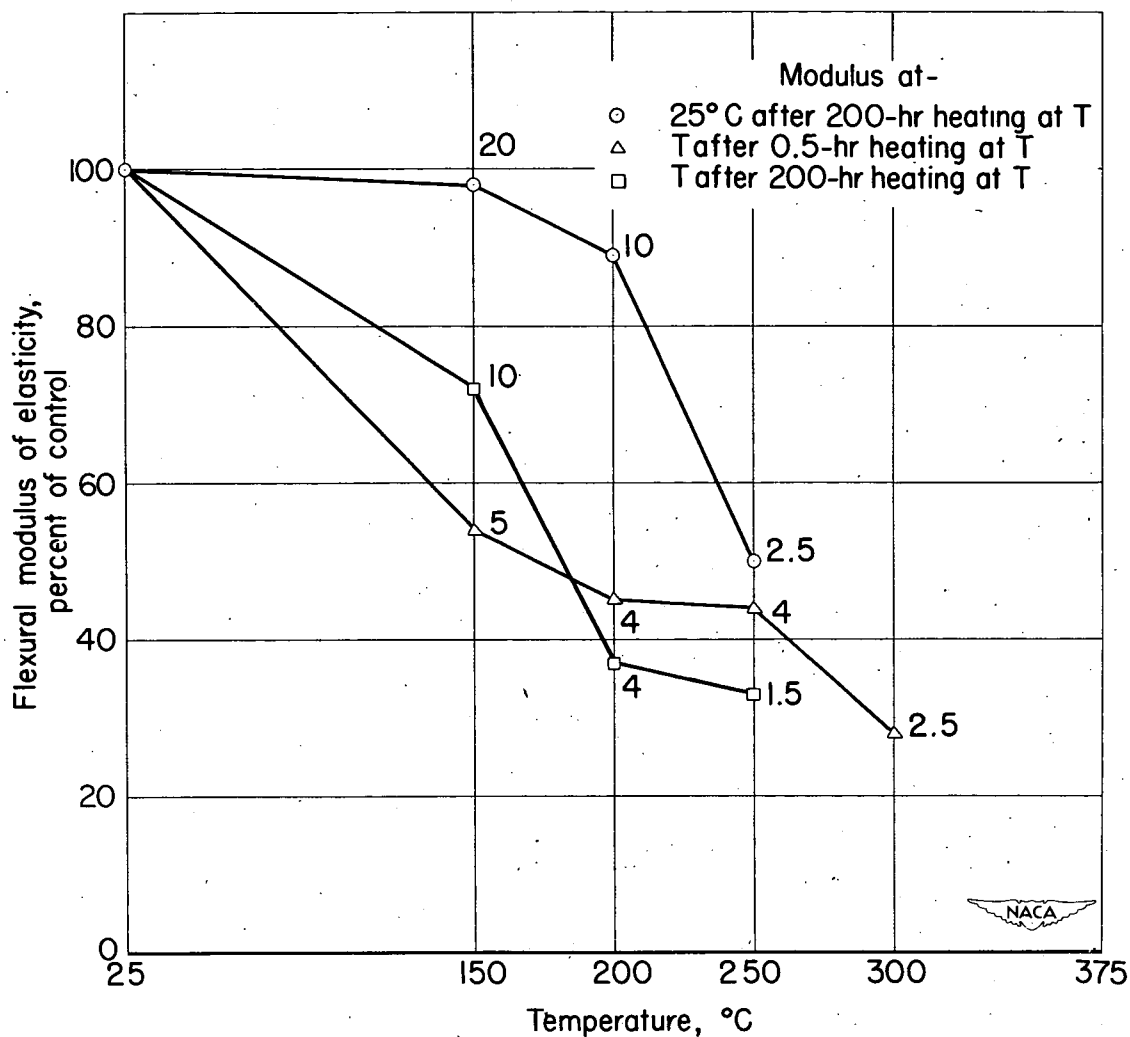


Figure 16.- Flexural secant modulus of elasticity of unsaturated-polyester laminate A for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point.

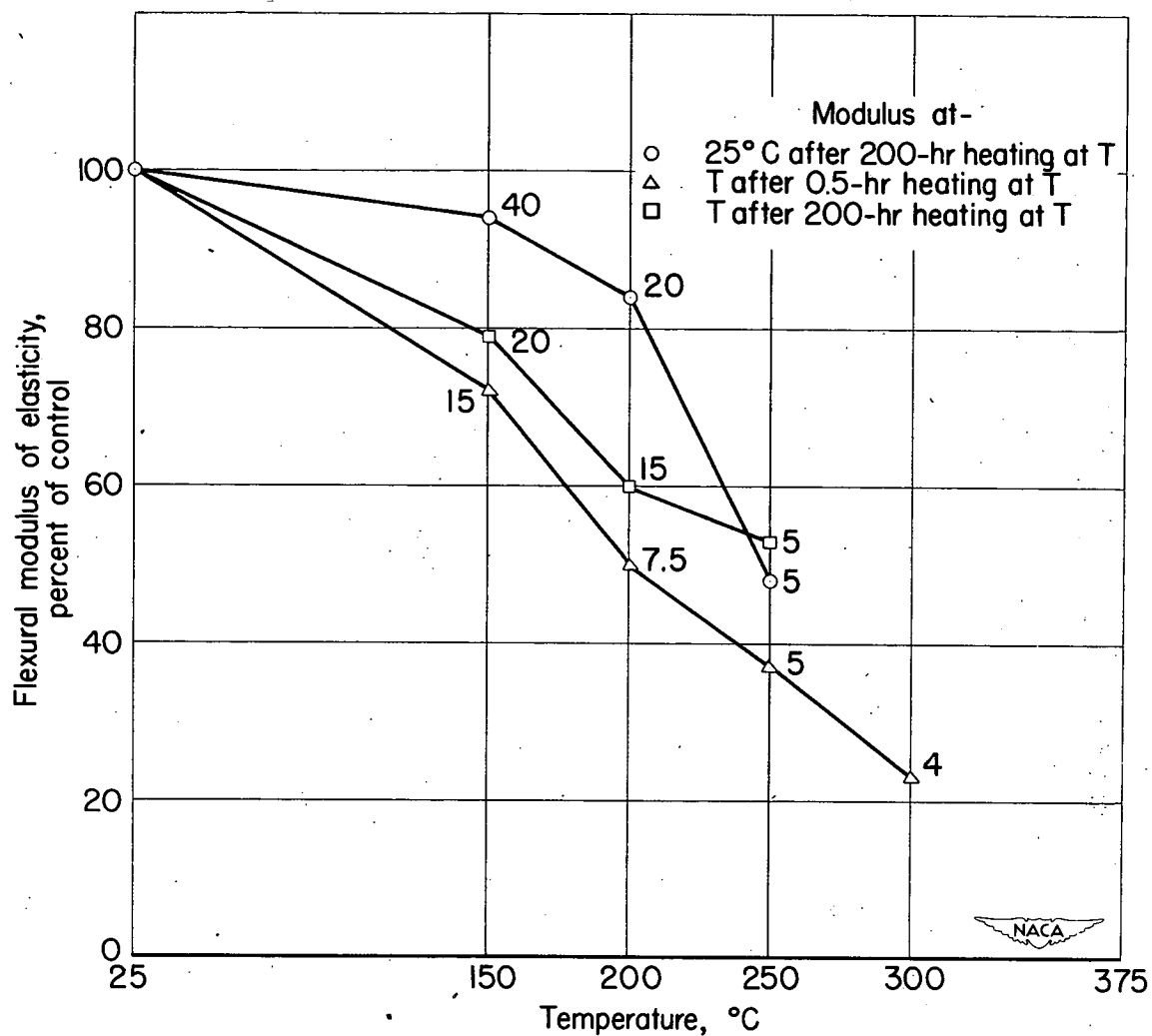


Figure 17.- Flexural secant modulus of elasticity of unsaturated-polyester laminate E for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point.

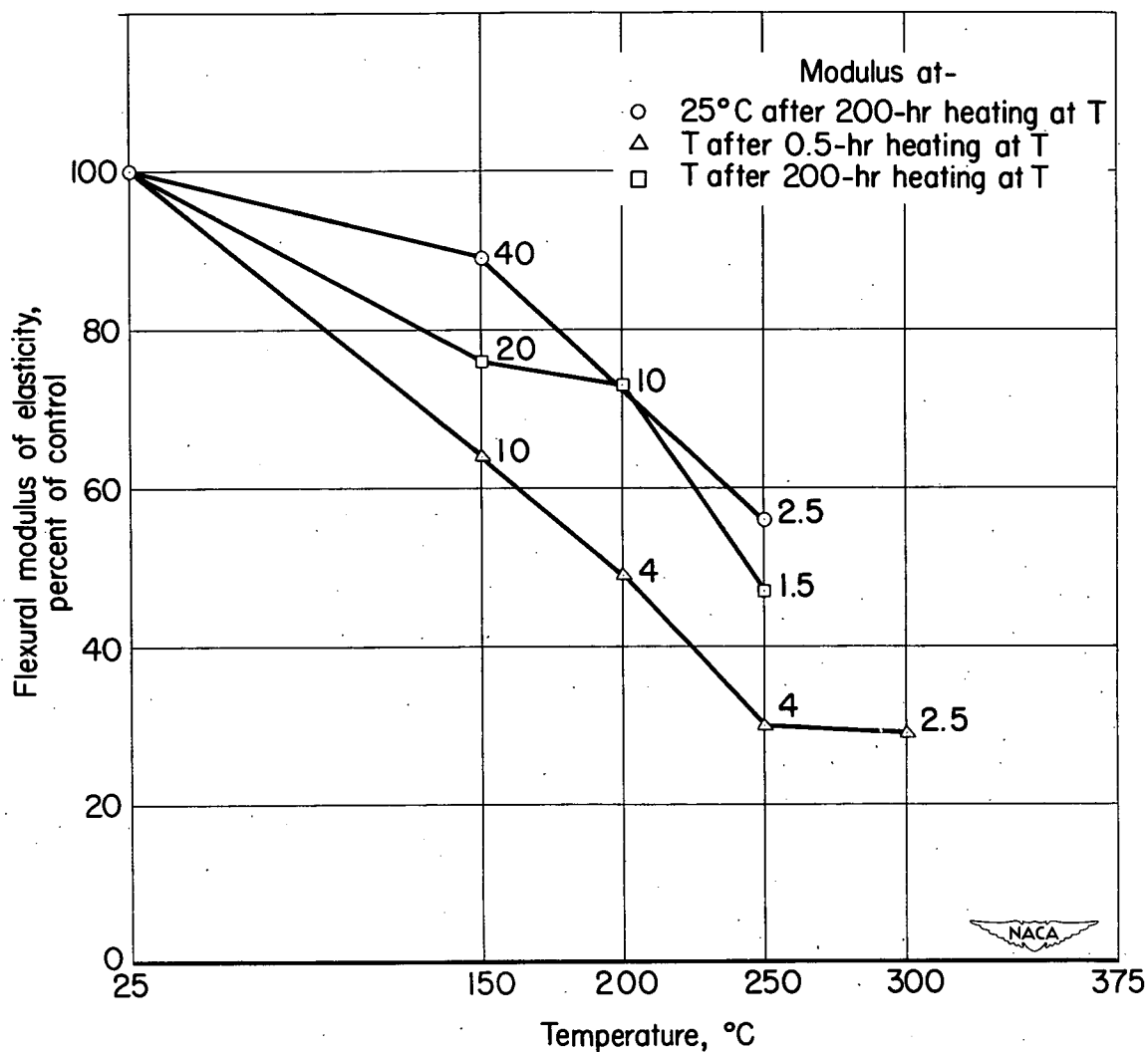


Figure 18.- Flexural secant modulus of elasticity of unsaturated-polyester laminate F for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point.

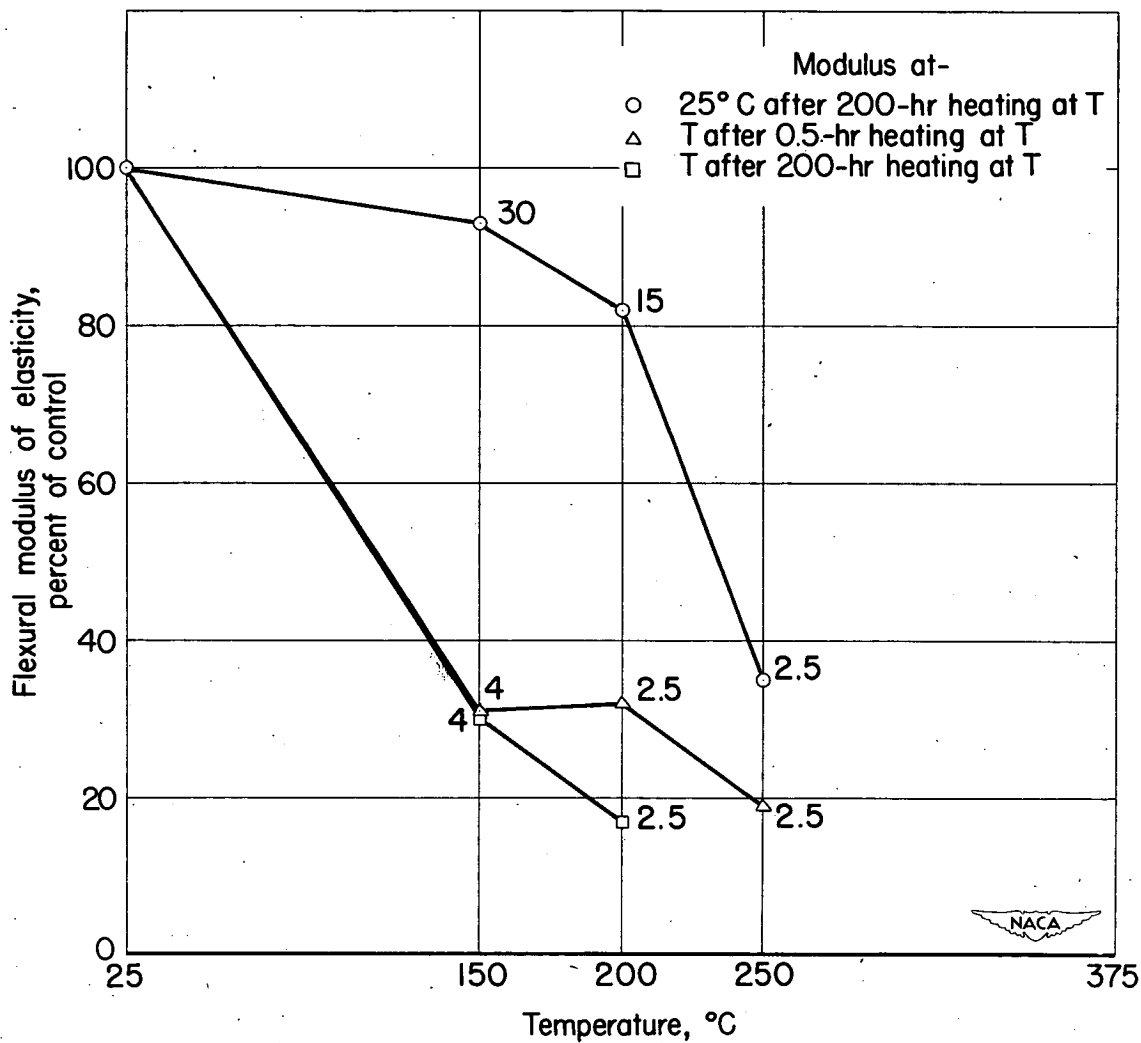


Figure 19.- Flexural secant modulus of elasticity of unsaturated-polyester laminate G for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point.

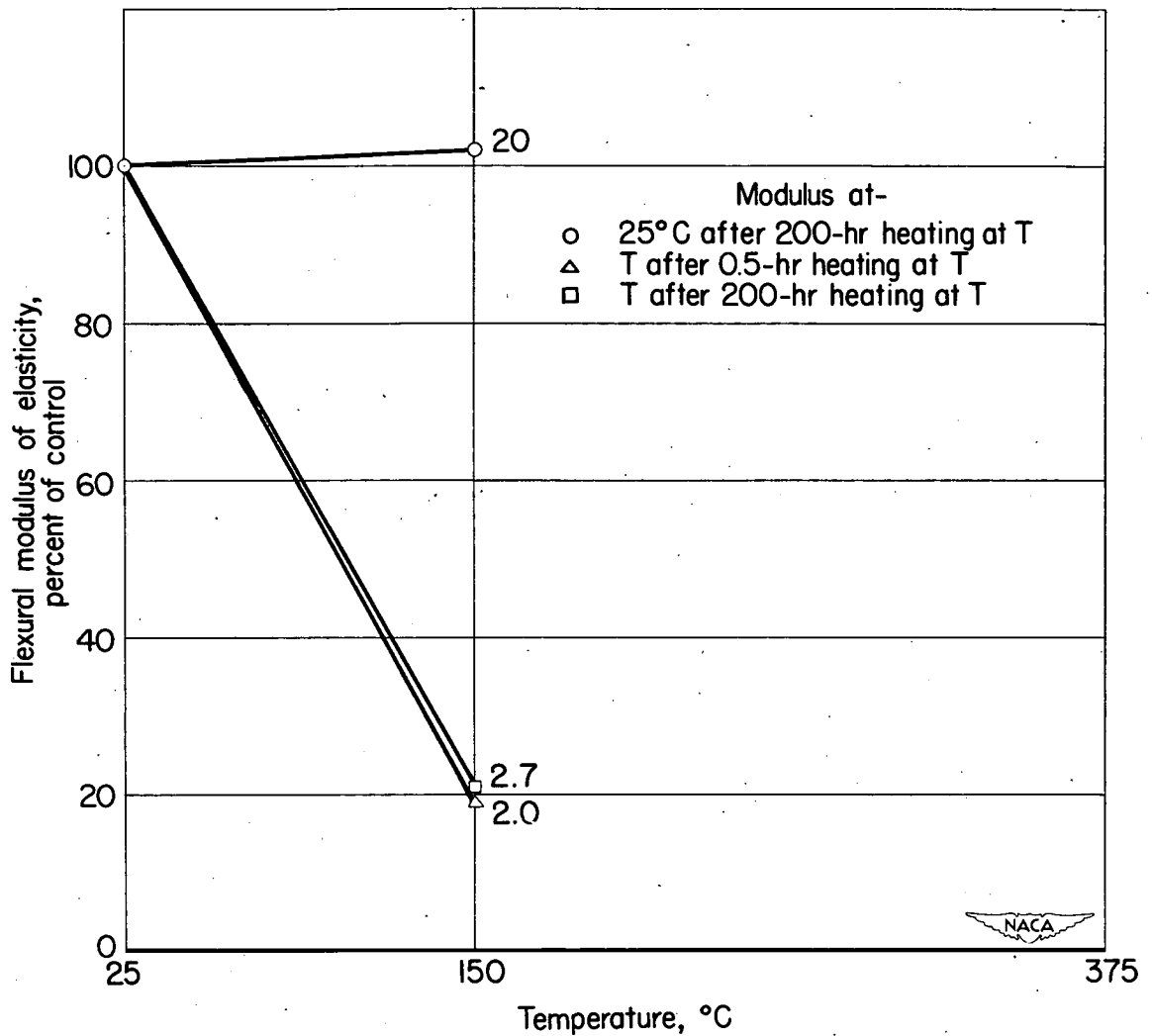


Figure 20.- Flexural secant modulus of elasticity of acrylic laminate C for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point.

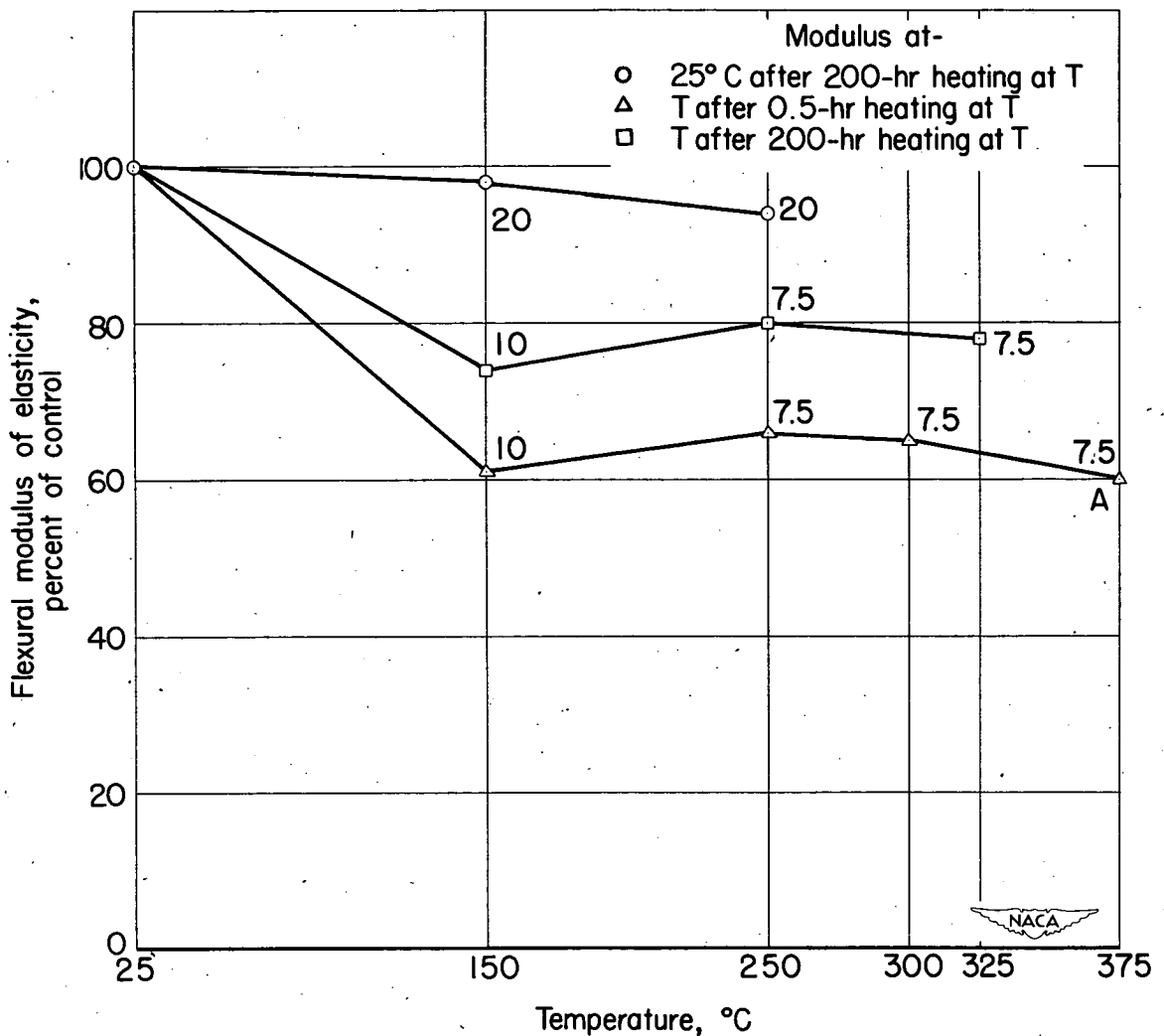


Figure 21.- Flexural secant modulus of elasticity of silicone laminate B for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point. A, result of test in which specimen tray was covered with asbestos paper.

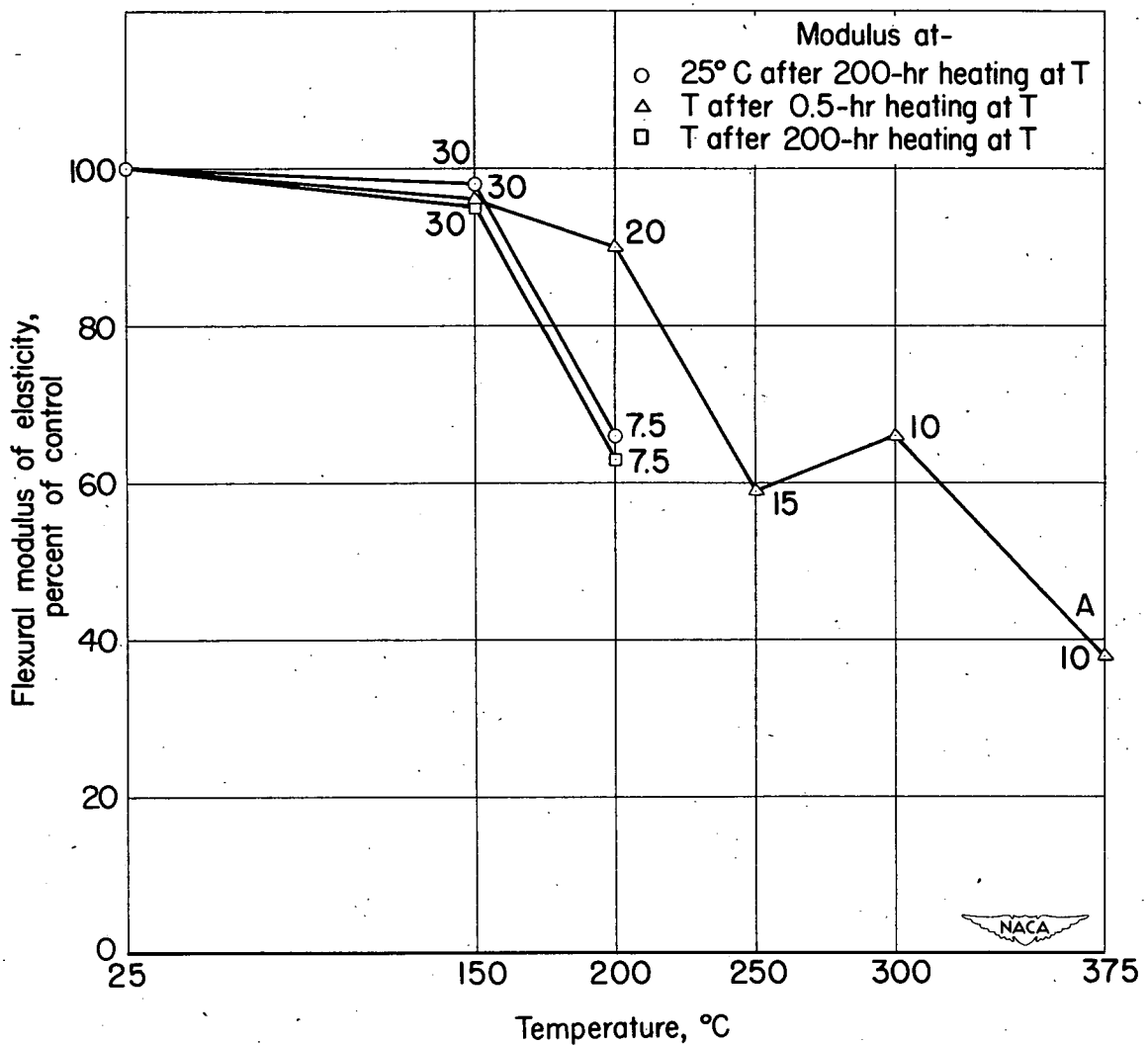


Figure 22.- Flexural secant modulus of elasticity of phenolic laminate H for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point. A, result of test in which specimen tray was covered with asbestos paper.

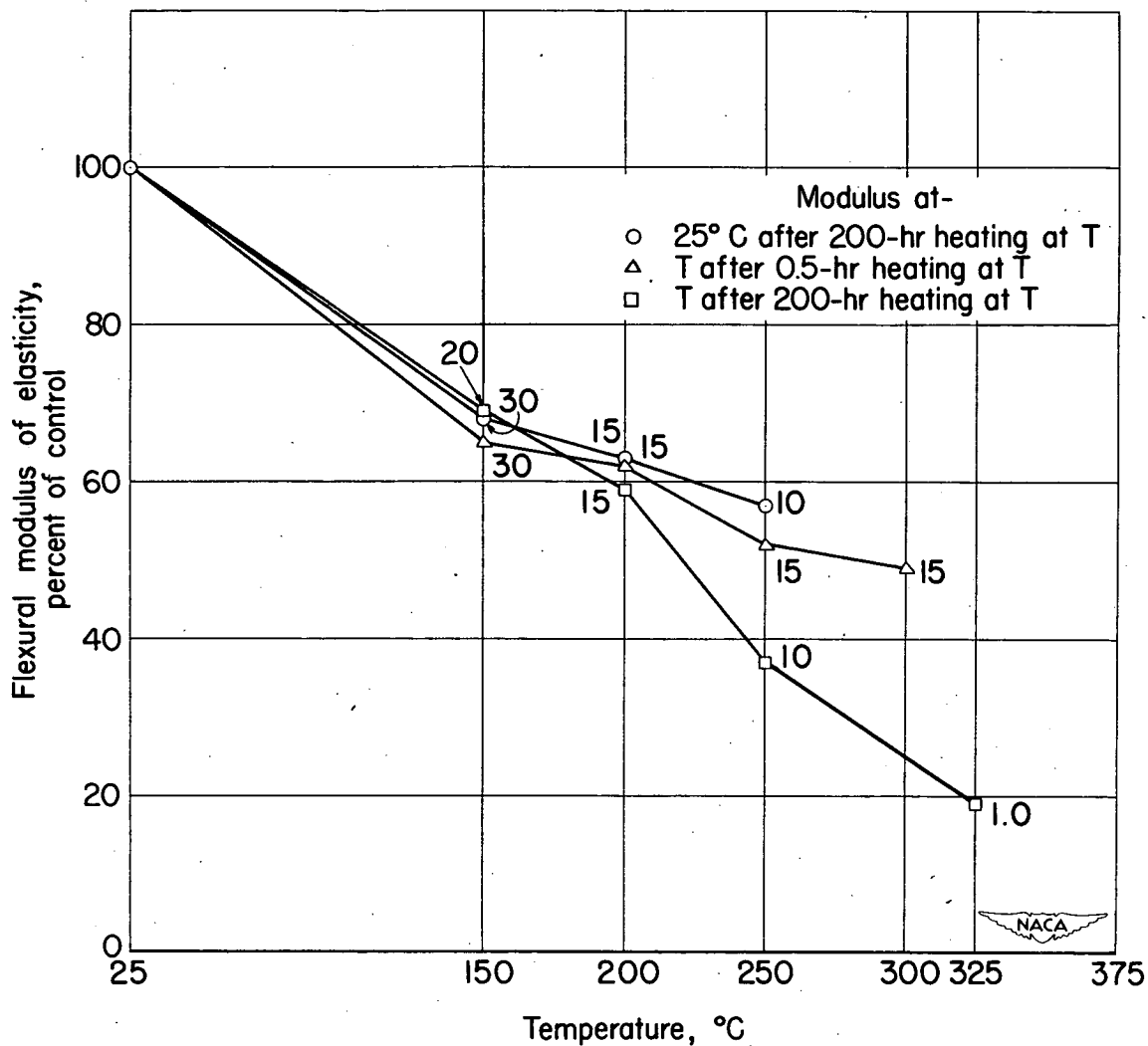


Figure 23.- Flexural secant modulus of elasticity of melamine laminate J for various conditions of heating and testing. Stress range was zero to stress in 10^3 psi indicated by number beside each point.

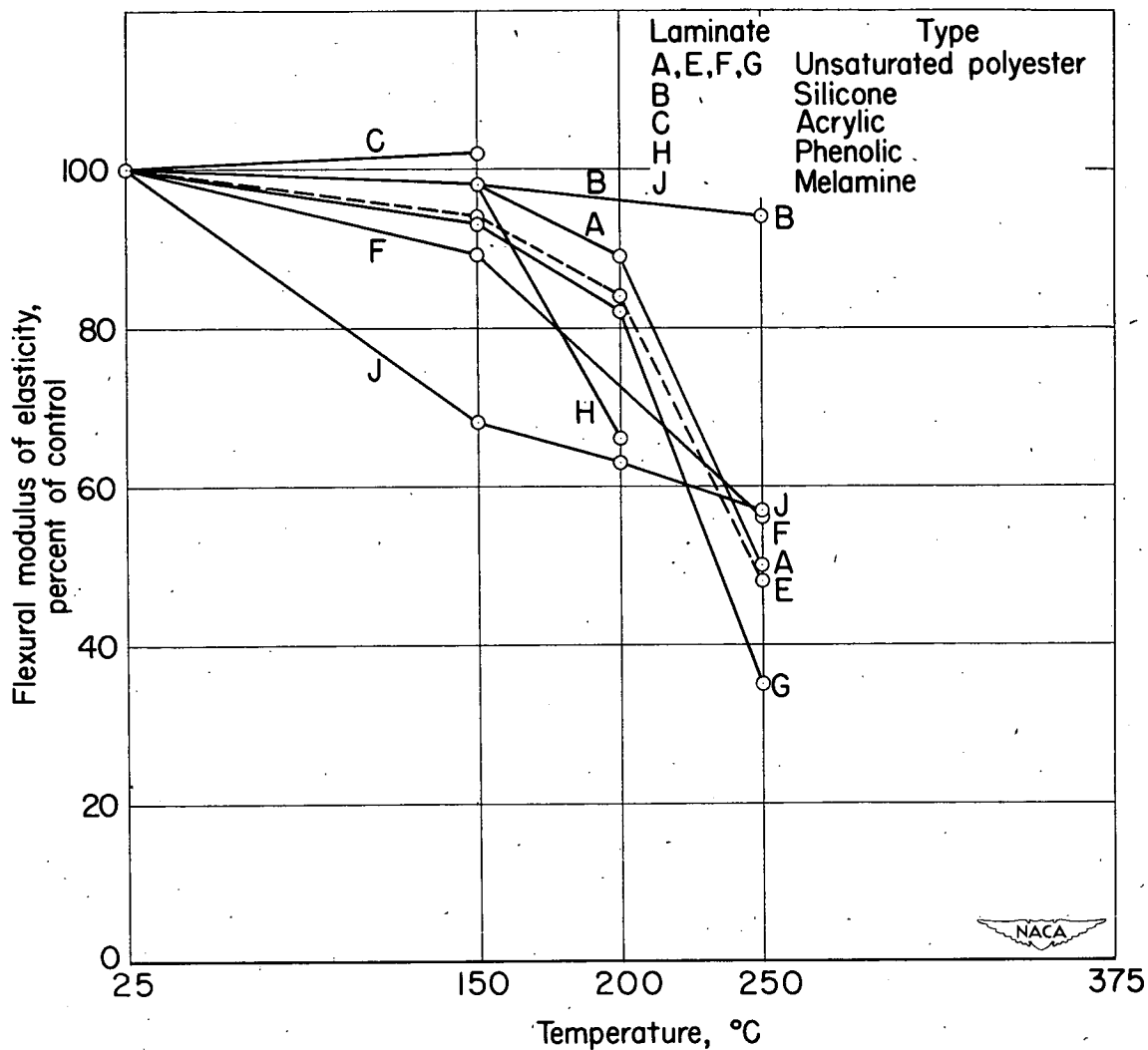


Figure 24.- Flexural secant modulus of elasticity of laminates at 25° C after , 200-hour heating at various temperatures. (Data taken from figs. 16 to 23.)

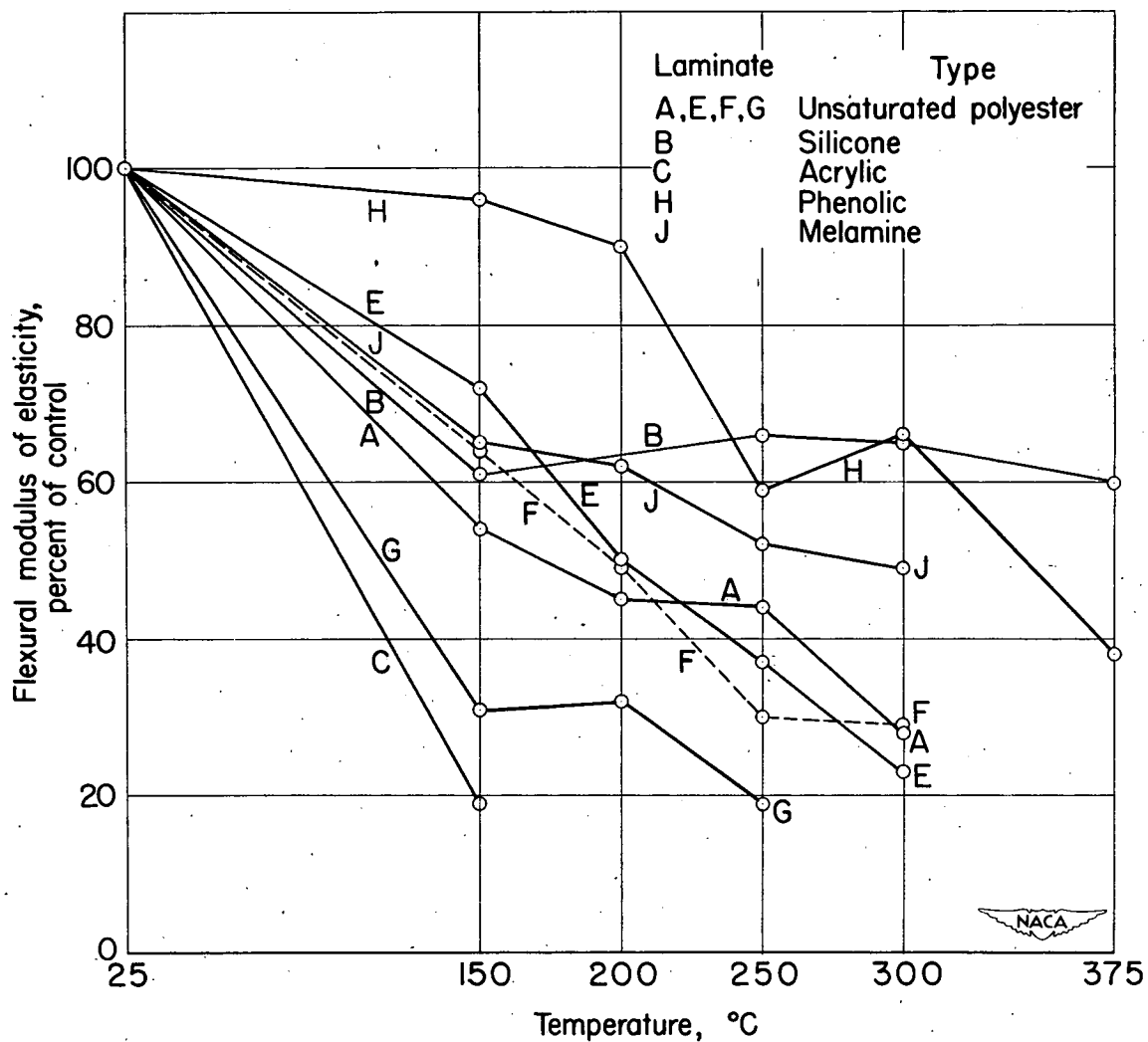


Figure 25.- Flexural secant modulus of elasticity of laminates at a temperature T after 0.5-hour heating at T . (Data taken from figs. 16 to 23.)

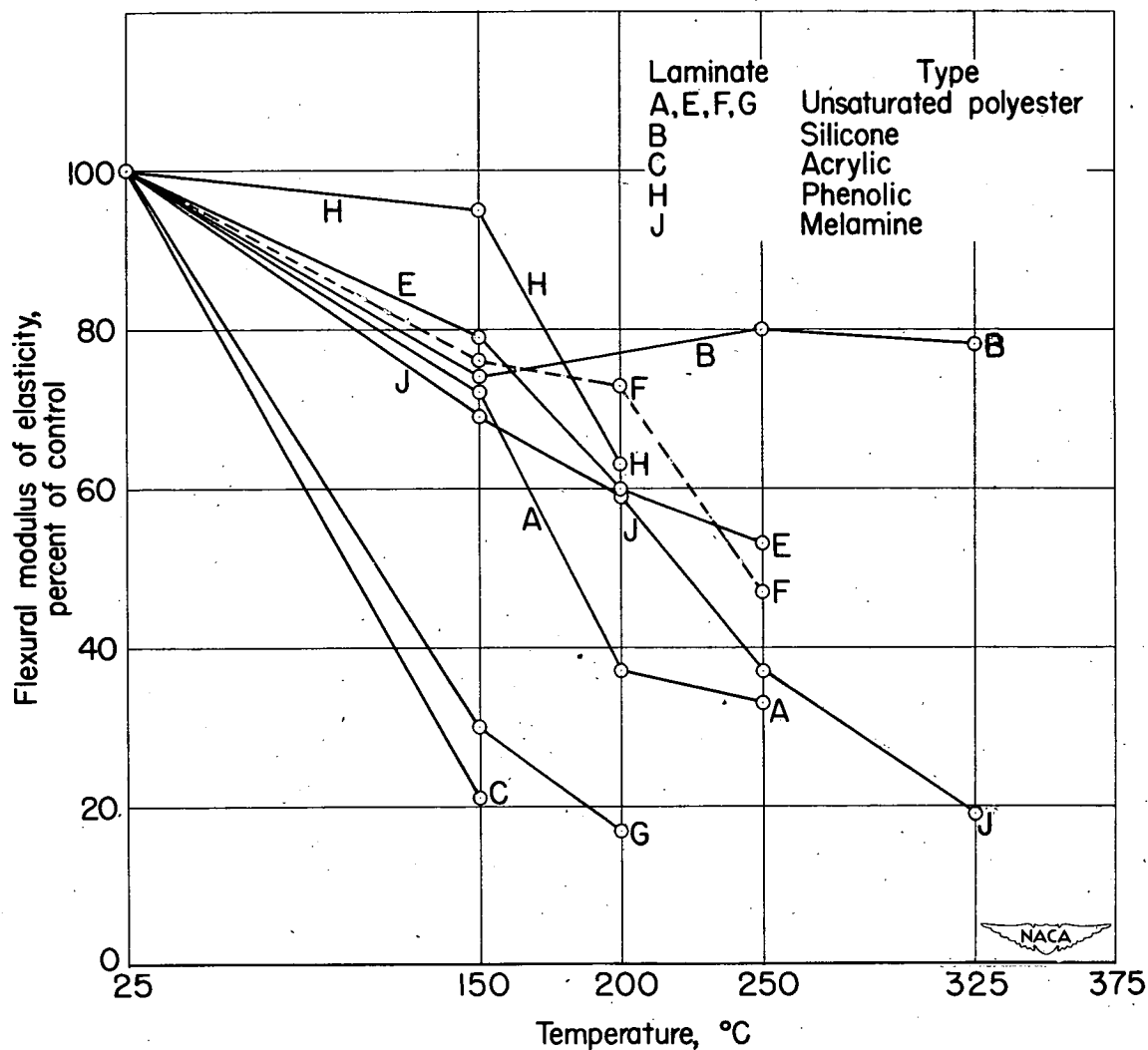


Figure 26.- Flexural secant modulus of elasticity of laminates at a temperature T after 200-hour heating at T . (Data taken from figs. 16 to 23.)

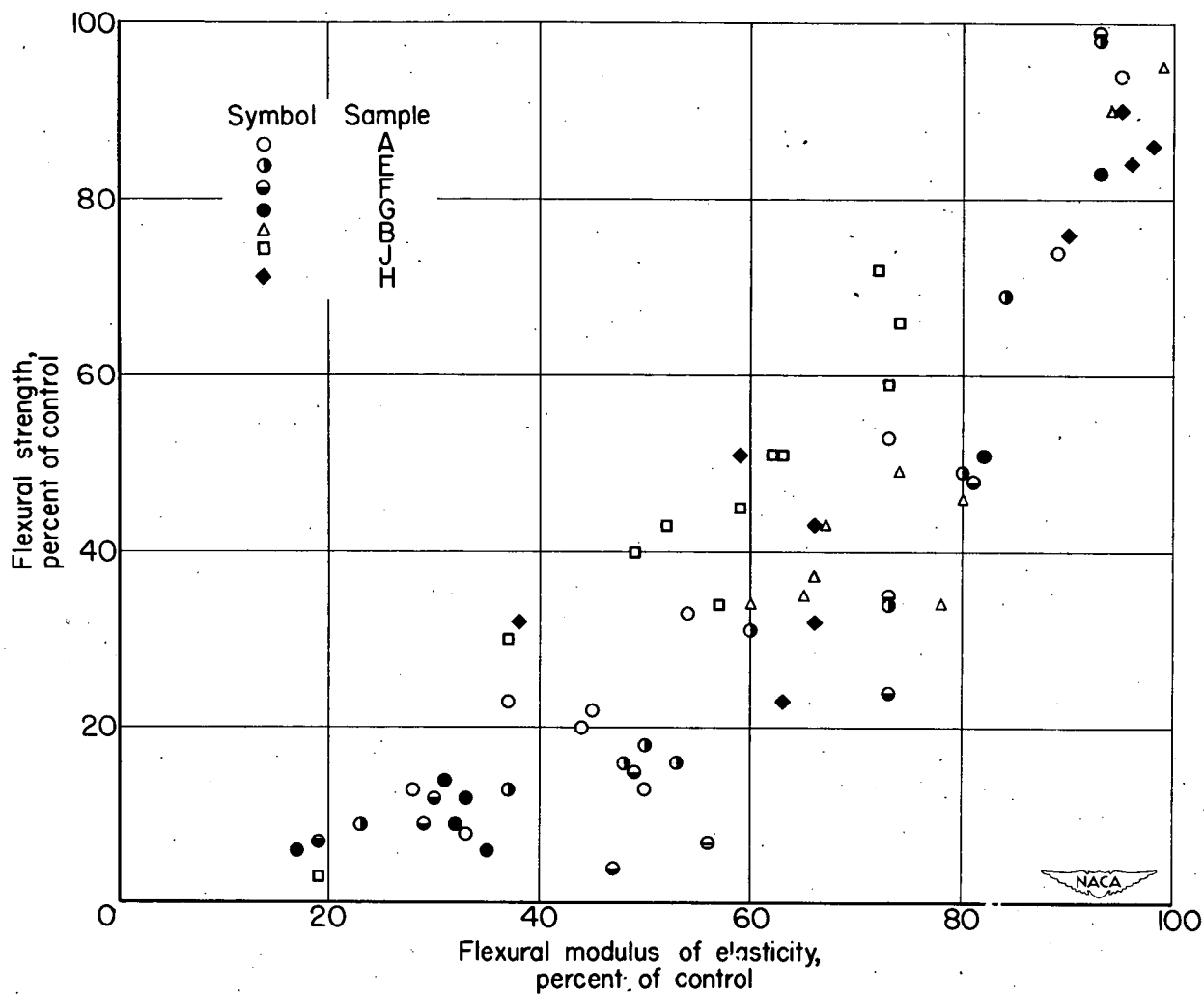


Figure 27.- Correlation between flexural strength and flexural secant modulus of elasticity. (Data taken from tables 2 and 3.)